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LIME, MORTAR, & CEMENT:

THEIR CHARACTERISTICS AND ANALYSES.

WITH AN ACCOUNT OF

ARTIFICIAL STONE AND ASPHALT.

BY

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GENERAL

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INTRODUCTION.

THE following short account of the character and

ADDENDA AND CORRIGENDA.

On page 20, 6th line from bottom, read "Arènes" instead of "arcues."

On page 22, 2nd line from bottom, read "Andernach" instead of "Audernach."

On pages 22 and 23, read "pozzuolana" instead of "puzzuolana."

Page 49, 2nd line from the bottom, should read: "B = the total weight of the chlorine found in the chlorides. The"

of the setting of mortar and cement, a question which is not yet finally solved. The various theories are discussed in the chapter relating to that subject, and some general suggestions made for further consideration.



INTRODUCTION.

THE following short account of the character and chemical composition of the various materials used in making mortars and cements, and the methods and results of their chemical analysis, has been compiled to meet the requirements of those who in the course of their technical experience have hitherto been at a loss where to look for guidance.

The author's experience has shown the necessity for recognised methods of examination and authentic results, which may be looked upon as a basis in the disputes which constantly occur in regard to the purity and strength of these materials.

In the following pages an account of the various limes will be found, the importance of which alone is very far reaching. By the detection of improper ingredients, and the establishment of a proper specification, the author was enabled on one occasion to remedy very grave defects, and to effect a saving to one firm alone of no less a sum than £2000 per annum! The various kinds of clay and sand, and their substitutes, are also fully dealt with.

Much dispute has arisen at various times as to the cause of the setting of mortar and cement, a question which is not yet finally solved. The various theories are discussed in the chapter relating to that subject, and some general suggestions made for further consideration.

The analytical question has been, the author trusts, fully met, and the chemical and physical factors of the problem clearly set out in detail. The results have been obtained only after much careful work, in connection with which sincere thanks are due to the painstaking work of Mr. R. Grimwood, F.I.C., official Chief Assistant in the Chemical Department of the London County Council.

The rough technical tests of mortar have been found by the author to be of no little value when time presses, and a general idea of the quality of the sample is required at short notice.

The mechanical tests for cements in particular are fully described, as no work of this kind would be complete without such a description, the author, however, does not claim any special features for these, which are so well known, but doubtless the opinions of the various experts quoted will be found of great assistance.

It is with peculiar pleasure that the results of the exhaustive researches of the author's former colleague, Mr. John Grant, C.E., on the testing and use of Portland cement, are included. It is unnecessary to do more than to refer to these classical researches to ensure their due share of attention.

The adulteration of Portland cement has of late years been brought prominently to the front owing to the position taken up by the London Chamber of Commerce, which has spoken plainly on the question. In this connection the researches of Messrs. Stanger and Blount will be studied with no little interest.

The elaborate experiments on the strength of brickwork

undertaken by a Committee of the Institute of British Architects need little recommendation. This invaluable work, by the kind permission of the Institute, has been carefully summarised in the following pages.

The use of concrete has now taken such a place in the engineering work of the day, that no apology is needed for the inclusion of a chapter on that interesting subject. Following this will be found an account of the various artificial stones now so largely employed.

The author is greatly indebted to Mr. Clifford Richardson, of Long Island City, New York, for his kind permission to insert copious extracts from the account of his valuable researches on the nature and origin of asphalt. Probably no other work contains such an exhaustive and reliable account of this valuable material.

The author has to express his sincere thanks also to Mr. Messrs. Stanger and Blount, Mr. C. Chambers Smith, C.E., Mr. R. Grimwood, F.I.C., Mr. R. G. Grimwood, A.I.C., Mr. Searles-Wood, F.R. Inst. B.A., and many other friends, for their assistance and kind permission to embody much material, without which the work would have been of far less value than it is hoped it will be.

It will be observed that the work has been condensed as far as possible with a view to greater convenience for ready reference.

CHAPTER I.

CHARACTERISTICS OF VARIOUS LIMES.

LIME in its pure state is the oxide of calcium. It is white, caustic, soluble in water to the extent of 11 grains in a pint, or 88 grains per gallon, and it combines

with carbonic acid to form carbonate of lime, which is familiar to us in an impure state in the form of chalk. The metal calcium is never met with in an uncombined state in nature, being always in combination, chiefly existing as carbonate. The "atomic" weight of the metal is forty, compared with hydrogen taken as unity. In its pure state it is yellow, harder than lead, ductile and mal-



A CHALK QUARRY.

leable. It combines with oxygen slowly in dry air, more rapidly in the presence of moisture, and must be preserved out of contact with air. When heated in air to a bright red heat it fuses and ignites, burning with a bright white light, and forming the oxide of calcium. Calcium oxide is the pure substance of our inquiry, but, as may be supposed, it is never obtained absolutely pure in commerce—in fact, for the purpose of making mortar and

cement it would be of but little use unless it had mixed with it other materials, such as silica, iron, and alumina.

In the form of carbonate, calcium forms a principal portion of some of the most common minerals and rocks, viz., arragonite, calc-spar, chalk, limestone, marble. It is a constituent of dolomite, which is a compound of carbonate of lime and carbonate of magnesia, having the composition $\text{CaCO}_3 \text{ MgCO}_3$. Calcium also occurs as sulphate in anhydrite (CaSO_4), in gypsum, alabaster, and selenite ($\text{CaSO}_4, 2 \text{ H}_2\text{O}$); as fluoride, in fluorspar (CaF_2); a phosphate in apatite [$\text{CaF}_2, 3 \text{ Ca}_3 (\text{PO}_4)_2$]; and as silicate is widely diffused in different minerals. Many of the salts of lime are normal constituents of soils, plants, and of the animal body; egg shells, coral, and the shells of the mollusca consist chiefly of calcium carbonate, whilst the mineral matter of bones consists chiefly of calcium phosphate. The carbonate (held in solution by free carbonic anhydride, better known as carbonic acid) and the sulphate are also normal constituents of natural waters, and cause, with the magnesia and other salts, the property known as hardness.

If a fragment of white marble, which is practically pure carbonate of lime, be heated in such a manner that the carbonic acid set free can escape, a white substance will be obtained, which is the oxide of calcium, or quicklime, having the composition, chemically expressed, of CaO , thereby indicating the combination of one atom of calcium with one atom of oxygen, and this compound has a "molecular" weight of 56, being 40 for the calcium and 16 for the oxygen. Its specific gravity is 3.08, water being taken as unity.

This white, amorphous solid attracts water and carbonic acid from the air, and under favourable conditions combines

with water with great energy and much evolution of heat, forming calcium hydroxide, according to the equation, $\text{CaO} + \text{H}_2\text{O} = \text{Ca}(\text{OH})_2$. When water is poured upon quicklime, the latter swells up, cracks, and emits steam, and finally falls into an impalpable powder. The purer and fresher the lime the more rapidly does this phenomenon occur. In this condition the lime is known as "slaked." If an excess of water has not been employed, the powder is to all appearance dry, and is far finer than the unslaked lime can be obtained by any process of grinding. When in the course of time the lime again combines with carbonic acid by absorbing it from the atmosphere, it necessarily parts with the water so taken up, the carbonic acid replacing it; thus, $\text{Ca}(\text{OH})_2 + \text{CO}_2 = \text{CaCO}_3 + \text{H}_2\text{O}$. This action is the cause of the efflorescence on brick-work known as "salting out," which will be referred to later on. The water thus displaced dissolves other salts, and the solution formed finds its way to the surface of the work, where the water evaporates, leaving the crystallised salt as a crust on the surface of the wall. From the above observation it will be seen that lime, considered as a chemical substance, is a very definite body, having characteristics peculiar to itself, and that these characteristics are the same wherever the lime may have been obtained. It is when we come to consider the actual properties of the various limes obtained in commerce that certain very specific characters are brought into play, and it is these which we have to consider in relation to the adaptability of the commercial lime for certain uses. Thus the lime most suitable for making mortar is by no means the best for making lime-water in large quantities, or for the purpose of the gas manufacturer in purifying his gas. These differences, it will be seen, are due to certain substances—or impurities, strictly speaking—which affect the quality of the lime.

The following table showing the approximate composition of some typical limestones, and the percentage quantities of pure lime and magnesia in the properly burnt stone, will

Table of the approximate composition of various Limestones, and the percentage of pure Lime and Magnesia in the Burnt Stone.

Limestone.	Unburnt Stone.			In Burnt Stone.	
	Carbonate of Lime.	Carbonate of Magnesia.	Clay, &c., and Moisture.	Lime CaO.	Magnesia MgO.
PURE, OR "FAT," LIMES —					
Marble	100	—	—	100	—
White Chalk	98½	0½	1	98	0½
Oolite	95	2	3	95½	1½
POOR LIME —					
Silicious Oolite	70	3½	26½	73½	3½
FEEBLY HYDRAULIC—					
Grey Chalk	92	—	8	86½	—
HYDRAULIC—					
Dolomite	51	40	9	53½	36
Carboniferous	86	—	14	81	—
Grey Chalk	83	—	17	73	—
STRONGLY HYDRAULIC—					
Blue Lias	79	—	21	72	—
Carboniferous	71½	1½	27	60	1
Scotch	68	1	31	56½	0½

serve to indicate the differences in character of the raw material before it is converted into lime by being burnt in the kiln. First of all, it is to be noted that the various

limestones may be classified under four heads, viz. :—(1) Fat or pure limes ; (2) poor limes ; (3) hydraulic limes ; (4) cements, natural and artificial. As it will be seen, these classes merge gradually into one another, and it is difficult to draw a sharp line of demarcation in many cases ; therefore we have “good” and “bad” limes, according to the purpose for which they are intended. These analyses may be accepted as forming an accurate sequence representative of the limes in question.

From this table it will be seen that the hydraulic character of some limes is due to the presence of certain impurities, viz., silica, iron, and alumina, which are included under the head of “clay” ; and that as these increase in quantity up to the extent of about 30 per cent. the “hydraulicity,” as it is called, of the lime produced by the burning of the stone is increased.

It will now be seen how important it is that the use to which the lime is to be put should be first considered before a purchase is made. Thus, if the lime is required for use in a gasworks, &c., where the chemical activity of the lime is of the first importance, it should be as pure as possible, and therefore “fat” limes should be obtained. If, on the other hand, the lime is required for mortar making, good hydraulic lime should be purchased. In the case of the manufacture of cement, the quality of the limestone employed must be considered in regard to the quantity of clay to be mixed with it to form the “slurry,” from which the cement is manufactured. This point serves to indicate the importance of chemistry in regard to our manufactures. For years the English Medway cements were considered far superior to that of German manufacture, because the ingredients naturally to hand constituted the best materials for the purpose ; but as soon as the German manufacturers

studied the question from the chemical standpoint they were enabled to so choose their ingredients as to turn out cement equal to the best of the English makes.

It will be understood that when the limestones are burnt the relative proportions of lime, &c., vary by reason of the loss of the carbonic acid which is driven off in the process. Thus, while the marble would show still 100 per cent. of pure lime, there being no impurity present, the white chalk would yield a lime having the composition of:—Lime, 55·2; clay, 1·2 = 56·4; or, expressed in percentages, lime, 97·9; clay, 2·1. In like manner the carboniferous limestone, having 86·2 per cent. of carbonate of lime, would, by the loss of carbonic acid, yield only 48·3 of lime to 11·2 of clay the percentages being:—Lime, 81·2; clay, 18·8. It will be remembered, from what has been stated in the first part of the chapter, that each 100 parts of carbonate of lime yields, if thoroughly burnt, only 56 of lime, by reason of the loss of 44 parts of carbonic acid.

The following specification for the supply of lime has been found by the author to fully answer its purpose, and may usefully be inserted here:—

GENERAL CONDITIONS.

1. The lime is to be delivered by the contractor at the works, alongside the wharves or jetties of the purchaser, as may be directed from time to time.

2. Each parcel is to be sampled on delivery, and in the first instance examined by the purchaser. In the event of his analyses differing from those of the sellers, samples are to be submitted to a referee, whose report shall be binding on both parties. Should the contractor not be represented at the time of the sampling (of which due notice will be

given) the decision of the purchaser as to the quality of the lime must be accepted as final.

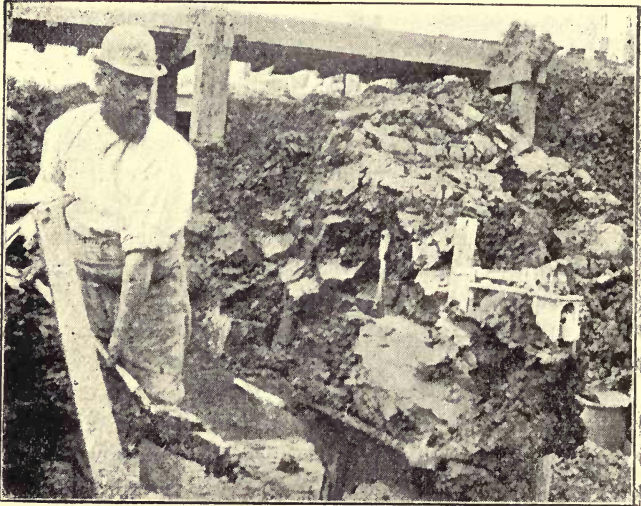
3. The lime is to be clean, well-burnt, hand-picked lime, free from dust, coke, and clinker. The price quoted by the tenderer is to be for lime containing 95 per cent. of actual caustic lime (CaO). The purchaser may reject any parcel of lime containing less than 85 per cent. of actual caustic lime, but may, if he think fit, accept the whole or any part of such parcel.

The price to be paid for any lime delivered and accepted, which contains more or less than 95 per cent. of actual caustic lime, is to be greater or less than the price quoted in strict proportion to the excess or deficiency of actual caustic lime, except that where the deficiency exceeds 10 per cent., the deficiency beyond 10 per cent. is, in fixing the price as aforesaid, to be considered as double what it actually is.

4. Samples of the lime proposed to be delivered, with the name of the source or district from which the same is obtained, must be submitted with each tender, which will otherwise not be considered.

CHAPTER II.

CLAY.



WEIGHING OUT THE CLAY.

By the process known as "weathering," already formed rocks are gradually disintegrated, and the fine mud thus formed constitutes the very variable materials classed for convenience under the generic term *Clay*. The principal ingredient is a silicate of alumina, which when perfectly pure is white. In this form it is known as Kaolin, or white-china-clay, pipe-clay, fuller's earth, &c. These correspond more or less to the composition Al_2O_3 , 2SiO_2 , $2 \text{H}_2\text{O}$. White marl, loam, &c., are classes containing various.

admixtures, such as calcium carbonate, oxides of iron, magnesia, &c.

Fire-clay, largely found in the proximity of coal seams, contains iron, and is nearly free from lime and the alkalis, soda, and potash. These are largely used for the manufacture of pottery, as at Stourbridge. The most refractory, used for the manufacture of articles which have to stand an intense heat, is composed of: Silica, 74 per cent.; alumina, 16 per cent.; ferrous oxide, 3 per cent.; alkalis, 1 per cent.; water, 6 per cent.; with traces of lime, magnesia, sulphuric acid, and chlorine.

Brick-clay is a term applied industrially to any variety of clay, loam, &c., used for making bricks and low-class pottery. It is very impure, containing large quantities of iron, &c. The following may be taken as its approximate composition, but various samples differ very largely:—Silica, 50 per cent.; alumina, 35 per cent.; ferric oxide, 8 per cent.; lime, 1 to 2 per cent.; magnesia, 5 per cent., &c. The colour of the bricks is due to the quantity of iron present, and the extent to which the bricks are burnt.

Mudstone is a fine sandy argillaceous rock, free from stratification, and harder than the usual clays.

Shale is a clay which has been deposited in thinly stratified layers. These are capable of being split into hard sheets or leaves. There is a great variety of these deposits having very different characters, according to the nature of the clay originally deposited, gradually passing into the slates.

In the technical terms of the brickmaker, pure clays composed of silicate of alumina, with but small quantities of lime, magnesia, &c., are known as "foul" clays. Loams, or "mild" clays containing sand, are known as "sandy clays."

Marl is a clay containing a large proportion of carbonate of lime.

Malm is an artificial imitation of natural marl, made by mixing clay and chalk in a wash-mill. It is generally known under the term "washed clay."

Pipe-clay is a greyish-white clay, greasy to the touch, and adheres strongly to the tongue. On being burnt it turns white. It is found in Devonshire, Dorsetshire, and on the Continent. It is used for the manufacture of clay pipes hence its name.

[A species of unctuous clay is eaten by the Ottomaques, a tribe near the Orinoco. This habit, doubtless originating in famine, occurs also in Brazil and other places. It is stated that the clay used has a milky and not disagreeable taste.]

Red Clay is distributed to an enormous extent over the bed of the ocean, and may be said to exist everywhere in the form of ooze, clay, or mud. It is generally of a brown colour, in consequence of the presence of the oxides of iron, with some manganese. It has not the same properties as fire-clay, as it fuses into a magnetic bead, due to the large quantity of iron and free silica, derived from the sedimentation of the skeletons of silicious organisms, such as the diatomaceæ, and volcanic minerals.

Burnt Clay is very largely used for many purposes. As a manure it has, in certain cases, considerable value, whilst it is in great favour with the builder and engineer for many purposes. Within the last few years the use of burnt clay has been extended to the sanitary world, being employed for filling the "Bacteria Beds," which are now so rapidly displacing the chemical and land treatment for the purification of sewage. The method of burning the clay is to first kindle a fire with suitable materials round a stout piece of

oak or other hard wood. The clay is gradually piled up round this, and sprinkled with fine coal slack or breeze. Care must be taken that the fire does not break through the surface, as in that case over-heating may result, unless the burnt clay is required for certain purposes, for which it cannot be too well burnt. The alternate layers of clay and breeze are gradually added from time to time until the heap may equal hundreds of tons. Constant attention is required to see that the firing is equally distributed, and kept well within the heap.

Probably few materials have contributed more to the commercial success of the English industries than the wealth of good clays of all kinds which abound in this country. Other materials may show a larger net return, but in nearly all these cases clay in one form or another has been employed, even if only for the buildings in which the various operations are conducted. To clay we owe our buildings, our furnaces, our railways and their embankments and bridges, our potteries. It is largely used by our paper-makers, and in an endless variety of the arts and industries, contributing in a multitudinous number of ways to our health, our comfort, and our necessities.

From the foregoing short account of the various descriptions of clay which exist, it will easily be seen how important it is to ascertain exactly the character of any sample which it is proposed to use for the manufacture of any special article. In the case of cement it is clearly of the greatest importance to ascertain that the right proportions of silica and alumina are present. If, for instance, the cement maker has only at his disposal a marly clay, it is useless for him to proceed as if he were using a "foul" clay, as the proportions of lime to silica and alumina would be very different.

In like manner a mild or loamy clay must have the proportions of limestone increased, or it may be that the quantity of sand present would be excessive, and thus render it unsuitable.

It is in this connection that the value of a properly conducted chemical analysis is felt. In these days of severe competition the manufacturer cannot profitably neglect the aid of science, which has aided so much those industries which have been controlled by those who are wise enough to avail themselves of the help which precise knowledge can give.

CHAPTER III.

SAND AND ITS SUBSTITUTES.

NEXT to oxygen, silicon is the most abundant and widely-diffused element known. It never occurs in the free state, but always in combination with oxygen as silica (SiO_2), either alone or in combination with other substances, thus forming a large series of minerals, of which the following are some of the natural forms in which it is found:—

Quartz	Opal
Amethyst	Jasper
Agate	Flint
Chalcedony	Sandstone
Onyx	Infusorial earth
Cornelian	

Quartz and amethyst occur in well-shaped crystals, belonging to the hexagonal system (six-sided prisms). Agate and chalcedony are a mixture of crystallised and amorphous silica.

The following compounds of silica may be broadly classed as aluminium and magnesium silicates, but most of them contain other elements, such as sodium, potassium, calcium, iron, manganese, &c. Their chemical constitution is very complicated, and it is very doubtful as to whether it is thoroughly known:—

Aluminium Silicates.

Beryl
Clay
Felspar
Garnet

Magnesium Silicates.

Asbestos
Augite
Hornblende
Meerschaum

Aluminium Silicates.

Jade
Lapis Lazuli
Mica
Pumice Stone
Slate
Topaz

Magnesium Silicates.

Olivine
Steatite (French Chalk,
Soapstone, &c.)
Talc

The mineralogical characteristics of these substances are of considerable interest. The quartz occurs in various forms, such as rock crystal, which is transparent and colourless; rose quartz, red inclining to violet blue; milk quartz, milk white, and slightly opalescent; cat's eye, enclosing asbestos, greenish white or grey, olive green, red, brown, or yellow; avanturine, enclosing mica, yellow, red, green, or brown; siderite, indigo or Berlin blue.

Common quartz, either crystallised or massive, of various colours, is a constituent of many rocks; ferruginous quartz, or iron flint, is often found with iron ores.

Flint is of various colours, from greyish-white to red or brown. It is semi-transparent, and has a flat conchoidal fracture. It is found chiefly in the chalk formations and river drifts. The mode in which flint is formed is assumed to be by the gradual accumulation of soluble silica upon the silica forms of minute marine organisms, as traces of these are almost always found in the flint nodules. Dr. Bowerbank was of opinion, from the prevalence of silicious spicules of sponges in the nodules, that all flints had for their primary nuclei the silicious framework of the sponges which flourished in the depth of the sea during the cretaceous period. Flints are found often in the chalk hollows in extensive layers of beds, such as that in the Chipstead Valley, in Surrey, where, it is said, traces of ancient workings have

been found, and where at this time large numbers of flint stones are still unearthed.

Sand is composed of either the simple fragments of quartz and flint which have undergone but little trituration by the action of flowing water in rolling the particles together, or similar fragments after they have been rubbed together for long periods, whereby the sharp edges have been ground down, and the particles become more or less rounded. In the former condition, the sand is known as "sharp" sand, whilst in the latter it is "water-worn" sand.

Pit Sand is of the former character, and is the best suited for making mortar, but it often is mixed with clay and other matters.

River Sand belongs to the water-worn variety, and is generally very clean, fine, and white.

Sea Sand is, by reason of the greater attrition which it has undergone, very round and smooth. Its use is objected to by some as giving rise to the phenomena known as "salt-ing out" after the work is finished. This is doubtless due to the sand not having been washed in fresh water to remove the salts with which the sea water is so highly charged, and which, drying with the sand, afterwards crystallise out on the face of the work.

In connection with the investigation as to the deposits in the river Thames by the Royal Commission presided over by the late Lord Bramwell, in 1883, Dr. Sorby pointed out the following beautiful method of differentiating the particles of quartz from those of flint in the sand of the river bed.

Quartz sand is made up either of entire crystals or of fragments of crystals, so that each particular grain has one certain optical structure. A grain of quartz sand examined by polarised light under the microscope will appear to be

black when the two prisms of the polariscope and the grain of quartz are in a certain position to one another. It is not shown on the field of the microscope, because the axes of depolarisation are parallel. As the quartz is rotated on its axes, without the prisms being altered in their position, it becomes brilliantly illuminated and coloured with all the prismatic colours, and as the rotation is continued these subside, and the quartz is once more in darkness; and this alternation of brilliant illumination and darkness continues as the rotation brings the axes of polarisation parallel or not with that of the prisms of the polariscope.

Flint is made up of a great many very small crystals, and when it is seen in polarised light looks mottled. The difference between flint and quartz corresponds to the difference between a mass of granite and a piece of coloured glass. On the whole, the flint would look mottled, with black and white all over. The bright colours are not seen, because the size of the individual crystals is not sufficiently great to produce interference of a sufficiently high order to give any colour. In a hundred grains or so, one might be met with which would cause doubt, but the differences between the quartz sand and flint sand is as well marked as the difference between sheep and goats.

In France a kind of sand exists in large quantities, to the extent even of hills, which consist of finely-divided chalk in grains of various sizes mixed with various coloured clays in different proportions. That is called "arcues," and is used with the rich loam from the rivers. Disintegrated Grauwacke rocks afford an argillaceous sand, consisting of quartz, schist, felspar, mica, &c., held together by clay. This is used both in its natural condition, and also after calcination, when it resembles puzzuolana, referred to later on.

Standard Sand for Cement Testing.—In consequence of the great variety in the results obtained in the tests of Portland cement with different descriptions of sand, it was found desirable to agree to some uniform method of procedure. This was pointed out by the late Mr. John Grant very clearly. In the course of his researches he found that “The washing of the sand seems to add about 35 per cent. to the strength at the end of a week; but at the end of a month this experiment is not so satisfactory. With washed pit sand, in the proportion of one of cement to two of sand, the breaking weight, at twenty-eight days, was 330 lb., or 46·2 per cent. above the first specimen of Thames sand, or 31·21 per cent. above the same sand when not washed.”*

The result of these and numerous other similar experiments was that a general agreement was arrived at to employ Leighton Buzzard sand, which is first carefully washed to remove all earthy matter, &c., and then sifted through a mesh of 400 to the square inch, and again through a mesh of 900 to the square inch. The portion of the sand taken as a standard for cement testing is that which is caught on the 900 mesh sieve.

Substitutes for Sand.—Whilst crushed flints or quartz, by reason of the sharp edges giving a better hold to the brickwork, is by far the best material to use with lime for making mortar, well-washed pit sand or river sand are the next best materials for the purpose. Many substitutes for these have been proposed. Chief amongst these may be placed well-burnt clay, either in the form of ballast broken down to a sufficient degree of fineness, or as broken bricks. The Romans are said to have used pounded tiles very largely, and the French are strongly in favour of it. When the

* “The Strength of Cement.” J. Grant. Page 8.

mortar was made with fresh water the results were very satisfactory, but when exposed to the action of sea water the work lasted only a few years. In England crushed burnt clay and bricks are very largely used, but unfortunately, the system of grinding all the materials together in a mill lends itself to the introduction of large quantities of earthy matter, which has a most disastrous effect on the subsequent strength of the mortar.

Well-burnt cinders or clinkers make excellent mortar, whilst furnace slag and the scoriæ from ironworks answer very well. The use of coal cinders from the domestic dust-bin is most strongly to be deprecated, unless these are passed through a "destructor" to destroy all the foul organic matter with which they are unfortunately so freely mixed. The author has examined many samples of mortar so prepared, which have been after a short time putrid and covered with fungoid growths. The use of such materials, unfortunately only too common with unscrupulous builders, should be made a criminal offence, punishable with imprisonment. To sacrifice the health of future dwellers in the houses so built for the sake of the small gain obtained by the use of this filthy matter is simply unpardonable.

The volcanic substance known as "puzzuolana," which is so common in Italy, has long been used with the best effect. This substance derives its name from Pozzuoli, near Naples, where it is found in great quantity. It is the basis of the best Roman mortars, both ancient and modern. It is shipped to England from Civita Vecchia. The colour of the material is either grey, reddish brown, or violet. It is roughly granular, resembling a cinder.

Similar deposits have been found in France, but the variety best known in England is that found near Audernach, on the Rhine, where it is known as tarrass or trass. One effect of

it is to render rich limes highly hydraulic, as well as improving the hydraulic limes. According to Vicat, mortars made with puzzuolana begin to set under water the first day, begin to get hard on the third day, and are as hard as bricks at the end of twelve months. The proportion in which it is used by the French authorities is 1 of lime to $2\frac{1}{2}$ of puzzuolana, or 2 of trass; or 1 of lime to 1 of sand and 1 of puzzuolana.

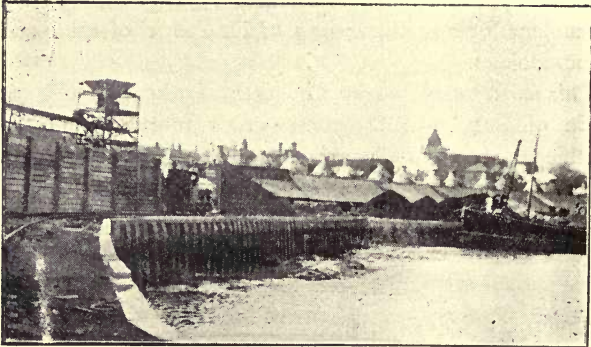
This substance is really a silicate of alumina, with some oxide of iron, and in some cases more or less lime. Berthier made an analysis of a sample of trass, and of one of puzzuolana, with the following results:—

	Trass.	Puzzuolana.
Silica	57'0	44'5
Alumina	12'0	15'0
Lime	2'6	8'8
Magnesia	1'0	4'7
Oxide of iron	5'0	12'0
Potash	7'0	1'4
Soda	1'0	4'0
Water	9'6	9'2
	<hr/>	<hr/>
	95'2	99'6

Smeaton, after a long series of experiments with these and other materials, employed them for mixing with lias lime to make a cement for use in the construction of the Eddystone Lighthouse.

CHAPTER IV.

MORTAR AND CEMENT.



HAVING considered the nature of the essential ingredients of mortar and cement, viz., lime, silica, and clay, it is necessary to consider the relation of these substances to each other in the production of the resulting compounds known as "mortar" and "cement." The use of cement in connection with concrete will be considered later on.

The essential feature in the formation of both mortar and cement is the hydration, or combination with water, of the lime, known as "slaking," and the subsequent "setting" of this material in contact with silica and clay. In the case of mortar we must have a hydraulic lime containing, roughly, about 3 parts of lime to 1 of clay. After slaking, this is added to a certain proportion of silica in the shape of sand. Other materials will answer to a certain extent, but, undoubtedly, good, clean, sharp sand is the best. The

proportion of lime to sand is generally very variable, and the only legal definition of "mortar" is that contained in the bye-laws of the London County Council, which specifies that the mortar to be used under Section 16 of the Metropolitan Management and Building Acts Amendment Act, 1878, must be composed of freshly-burnt lime and clean, sharp sand or grit, without earthy matter, in the proportion of 1 of lime to 3 of sand or grit. In the case of "cement-mortar," the cement to be used must be Portland cement, or other cement of equal quality, to be approved by the surveyor, mixed with clean, sharp sand or grit, in the proportions of 1 of cement to 4 of sand or grit. Also that burnt ballast or broken brick may be substituted for sand or grit, provided such material be properly mixed with lime in a mortar mill.

From the foregoing it will be seen that the composition of lime-mortar answers to that of Portland cement, when the latter is gauged with sand ready for use.

In the manufacture of Portland cement, lime and clay are mixed together, burnt, and ground into a powder. The quantities of these ingredients are not quite the same as those in hydraulic lime, the proportions being, roughly, 2 parts of lime (CaO) to 1 part of clay. The difference in the resulting compound is mainly brought about by the action of heat, by means of which the lime and silica of the clay are caused to combine, with the result that when moistened and allowed to set, the particular combinations so feebly present in the mortar are present to a much larger extent in the cement, with a consequent increase in its strength.

It will thus be seen that the art of the cement manufacturer consists in the selection of materials and mixing them together in such proportions as will produce a com-

pound having in its finished state the maximum strength obtainable. First, the quantity of lime (CaO) in the chalk used is ascertained by chemical analysis, then the quantity of carbonate of lime in the clay, if any, is determined. The presence of silica, as sand, in the clay is also looked for. Having ascertained these factors, the relative proportions of chalk and clay required to produce a mixture containing about 3 parts of chalk, or carbonate of lime if a limestone is employed, to 1 part of clay, are calculated, and a mixture is made by intimately grinding the two substances together. When this is done, a sample of the mixture must be analysed to ascertain whether the desired proportions have been obtained. If this be found not to be the case, either more chalk, or clay, as the case may be, is added, and the whole thoroughly incorporated and again examined. Unless these precautions are observed, the manufacture of Portland cement becomes a haphazard proceeding, with great risk of the production of an inferior article.

The desired mixture having been obtained, the mass is dried on large floors heated by the waste heat from the kilns. When thoroughly dry it is cut out in blocks and stacked in the kilns, which in many cases take charges of 40 tons. During the stacking fuel is mixed with the charge, and then the whole is burnt until the mass is at a glowing heat. Great care is taken that the mass is not overburnt, in which case the material is spoilt. When properly done the cooled masses have the appearance of a cinder. If the heating is carried to the extent of vitrification the cement is useless. The burnt cement is then ground to a fine powder, the strength of the cement depending very largely upon the degree of fineness. This was not clearly understood until the late Mr. John Grant, C.E., pointed out the

advantage of fine grinding. Since the publication of his results the matter has received much attention, with the result that greatly improved methods of grinding, such as the roller mill, have been introduced, and a consequently higher strength of cement obtained.

There is much difference of opinion as to the necessary degree of fineness. Mr. Grant specified that the residue on a sieve of 5800 meshes to the square inch (equal to about 76 per lineal inch) shall not exceed 15 per cent. by weight; whilst Mr. Michell proposed only 10 per cent. on a 50 sieve, equal to 2500 per square inch. Messrs. Hilton, Anderson, and Brooks supply, when required, a cement ground to 10 or 12 per cent. residue on a mesh of 32,000 = 180 per lineal inch.

The further question of the specification for Portland cement will be dealt with in the chapter relating to the 'Technical Testing of Cement.'

Roman Cement.—In 1791 Mr. James Parker took out a patent for the manufacture of so-called "Roman Cement." This he prepared by burning and then grinding the ferruginous nodules found in the Island of Sheppy.

This cement is used either alone or mixed with sand or other materials, as desired for the particular purpose for which it is to be employed. The following is the approximate composition of this material:—Silica, 15 per cent.; alumina, $5\frac{1}{2}$; oxide of iron, 7; magnesia, 1; manganese, $\frac{1}{2}$; lime, 45; moisture, carbonic acid, and loss, 26 per cent.

Similar cements to Parker's "Roman" cement are known as "Atkinson's," made in a similar manner in Yorkshire; whilst Medina cement is made from nodules obtained in the Isle of Wight. These were but the forerunners of Portland cement.

Selenitic mortar was a device of Major General Scott, who

mixed with the cement a substance containing sulphuric acid, namely, either plaster of Paris (sulphate of lime) or green copperas (ferrous sulphate). This had the effect of hastening the setting of the cement, but modern experience is against this addition in all cases where great strength is required.

Plaster of Paris.—This valuable substance is a sulphate of lime rendered anhydrous, or free from water, by heating. When in a crystalline condition it is known as selenite, largely used for the manufacture of polariscopic prisms, whilst in pure milky-white masses it is known as alabaster. The ordinary hydrated deposits, having the composition $\text{CaSO}_4 \cdot 2 \text{H}_2\text{O}$, occur in various rock formations, and are known as gypsum, which occurs largely in the proximity of Paris, whence the name of the cement made from it. There it is quarried very largely. The crude substance is heated in kilns to a temperature of 240 deg. Fah., then finely powdered. The heating causes it to part with its water of hydration, which begins to separate at about 175 deg. Fah. When used, the dry plaster is mixed with water, which rapidly re-combines with the anhydrous sulphate, and causes it to set.

Keene's Cement.—In consequence of the porosity and easily injured surface presented by plaster of Paris, many attempts have been made to harden it. For this purpose Keene heated the burnt gypsum with a solution of 1 part of alum in 12 parts of water, at a temperature of 95 deg. After standing in this solution for three hours, the gypsum is dried, re-burnt, and ground to a powder. This plaster is slower in setting than the ordinary plaster of Paris, but is about half as hard again.

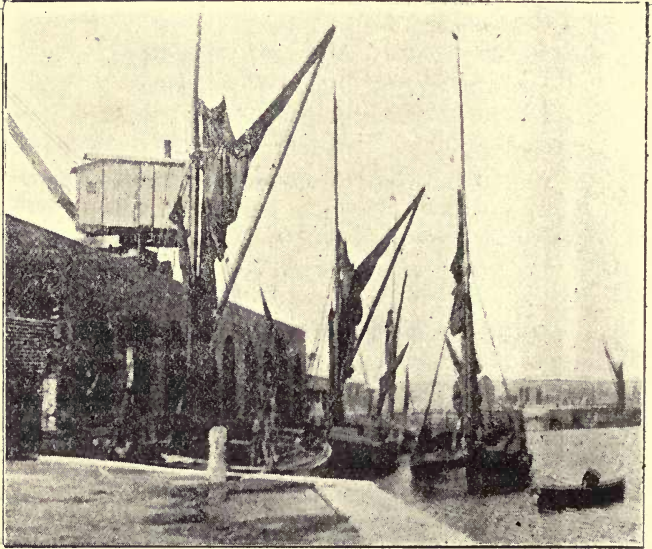
Parian Cement is plaster of Paris hardened with a 10 per cent. solution of borax.

Stucco is plaster of Paris prepared with a solution of glue.

Lime water has been recommended instead of fresh water for mixing with plaster of Paris ; also a solution of gum Arabic. Hardened plaster surfaces thus obtained may be stained and polished as desired.

CHAPTER V.

THE SETTING OF MORTARS AND CEMENTS.



As already shown in the previous chapters, mortar is essentially a mixture of hydrate of lime with three to four times its weight of sand, and according to the text-books its hardening is chiefly due to the gradual conversion of the lime into carbonate by absorption of carbonic acid, known strictly as carbon dioxide (CO_2), from the atmosphere. This absorption is at first rapid, and then gradually slower. Mortars even centuries old are said to still contain appreci-

able quantities of hydrate of lime. The hardening may be in some cases partially due to the formation of silicate of calcium, but the evidence of this is not very strong.

It is generally held that pure silica in the form of sand acts merely mechanically, and enters into no chemical combination with the lime. For all practical purposes this is true, but experiments have shown that in the course of several years some such action does take place to a very slight degree. Petzholt found :—

(1) That in mortar one hundred years old there was more soluble silica than in the original lime.

(2) That in mortar three hundred years old there was three times as much soluble silica as in mortar one hundred years old.

While these results may be accepted they yet leave the question open, as the slight action which had taken place was too small to throw any light on the process of hardening within the period of "setting." The degree to which lime is carbonated is also so slight within reasonable limits of time as not to warrant the assumption that the hardening properties are due to this action. Doubtless the re-arrangement of the molecules in what may be termed the initial stage of crystallisation, plays an important part, and from the fact that crystals of carbonate of lime adhere most firmly to the sand, and will stand a considerable degree of force before they can be released, it would appear that this mechanical action forms one of the most important facts in the problem.

It is well known that when lime is thrown out of solution by the process of water softening, introduced by the late Dr. Clark, the precipitated lime is generally in a crystalline form, and it is not unreasonable to assume that some such action takes place when mortar sets, the minute

crystals formed adhering more or less firmly to the grains of sand according to the conditions prevailing at the moment. This action would go far to explain the cause of the non-setting of mortar made with bad or imperfectly burnt lime, old mortar, &c. If the crystallisation of the lime takes place upon the surface of a substance which in itself is friable, the resulting mass will have a power of cohesion greater than that possessed by the same substance in proportion only to the power of the lime; just as the strength of a brick wall cannot practically be greater than the strength of the bricks used, as, however hard the cement, the bricks will give way under a strain in excess of the power of cohesion of their integral parts. Hence the absolute necessity for the "grit" of mortar to be clean, free from earthy matter, and of an unfriable nature.

This crystallisation theory also explains the reason why so many different substances may be used in making good mortar—a fact which the theory of chemical combination of the component elements is altogether unable to reduce to order.

Another point is, that the modern practice of grinding all the materials for making mortar together in a mill leads to a most unsatisfactory condition of the "grit." The crystallisation theory presupposes the presence of a hard and firmly resisting number of particles cemented together by the crystallisation of the lime, whether as carbonate, hydrate, &c. If, by grinding the grit to a powder, these "main supports," as the particles of solid matter may be termed, of the structure are broken down to a mere dust, then the initial power of resistance to pressure which they exerted in their unground state, will be thrown away, and a useless as well as dangerous muddy mass be obtained in their place.

Marignac has observed that anhydrous calcium sulphate

yields with water a supersaturated solution, which afterwards deposits crystals of the hydrated salt. With plaster of Paris heated at 140 deg., a solution can be obtained containing 9 grammes of the salt per litre, or four times the amount which exists in solution under normal conditions. Le Chatelier* considers that the setting of plaster of Paris is due to two distinct but simultaneous reactions. The anhydrous calcium sulphate when moistened with water dissolves and becomes hydrated, forming a supersaturated solution, and this supersaturated solution deposits crystals of hydrated calcium sulphate, which gradually increase in volume, and unite one with another. This progressive crystallisation continues so long as any anhydrous salt remains undissolved. This theory is supported by the fact that in practice 140 deg. is found to be the best temperature at which to heat the plaster of Paris, and Marignac found that the most highly supersaturated solutions are formed by calcium sulphate heated to this temperature. It is found that the addition of a small quantity of sulphuric acid or sodium chloride to the water used for moistening the plaster promotes setting. Both these compounds increase the amount of calcium sulphate which can exist in the supersaturated solution.

Similar phenomena of setting, due to deposition of crystals from a supersaturated solution, are observed on moistening coarsely-powdered anhydrous very soluble salts, such as sodium sulphate and carbonate, which readily form supersaturated solutions.

This theory is applicable to the setting of all mortars, especially cements and hydraulic mortars. The solubility of lime is well known. Le Chatelier has recently shown

* *Journal of Chemical Society*, July, 1882, p. 712.

that calcium aluminate is soluble, and he hopes to prove that calcium silicate is also soluble.

The character of the sand employed has also much to do with the result. I made an experiment some years back in conjunction with the late Mr. John Grant, whose work on cements is so well known and justly appreciated, in which two portions of a sample of good Portland cement were made up into briquettes with a given sample of sand. In the first the sand was in its normal state, but in the second experiment I heated the sand to redness, so as to completely oxidise the iron contained in it. The result was that the breaking strain of the briquette made with the heated sand was found to be nearly double that made with the unheated sand. Possibly, the greater combining power may have been due in some respects to an altered condition of the face of the particles of sand, in consequence of which the lime crystals adhered more firmly, and thus resisted the strain to a greater extent.

In consequence of pressure of other work, the experiments were not continued, but it may be that they would form the starting point of a series which might result in valuable information being obtained.

Now that means of obtaining excessive pressures are more readily accessible than formerly, it might be of value to undertake a series of experiments in which the briquettes made of various limes, cements, and sands, should be subjected to pressure of various degrees, and for various periods, and their breaking strain ascertained. Such a work might well be undertaken by a Committee of experts having the proper facilities at their disposal, as it is obvious the resources of a busy private laboratory are insufficient for the purpose.

The recent experiments on the breaking strain of brick

piers made with various cements, &c., by the Institute of British Architects might very well be extended in this direction. In the course of a series of experiments undertaken by a Committee of that body, a number of piers constructed of various descriptions of bricks and cements were kept for various periods, and then subjected to heavy pressures, and the results have been published in the form of two valuable reports, a review of which will be submitted later on.

The late Mr. John Grant, in his account of his researches on the strength of cements, presented to the Institute of Civil Engineers in April, 1871, remarks "that the cohesive varied directly as the adhesive strength of cement; that, in fact, neat cement was from three to four times the adhesive strength of any mixture of it with four or five times its bulk of sand; and on this account he was prepared to state in general terms that cohesive and adhesive strength might be taken as equivalent to each other. For the purpose of cementing together various materials, he need hardly point out that to have strong cement or mortar was not the only thing necessary. For instance, two cubes of glass could not be cemented together, however strong the cement, so that they should adhere with the same tenacity as two bricks or blocks of stone of a porous character and rough on their faces. It would not be possible, for example, to take such bricks as were recently used for lining the subway from the Houses of Parliament to the adjoining railway station, and to cement their highly glazed china faces together so that it would require the same pull to separate them as it would to separate two stocks, wire-cut gault, Fareham red, or other porous bricks, capable of absorbing one-fifth of their weight [of water. Whilst on this point, he might state that only second in importance

(if second) to the strength of the cement was that of seeing that the bricks or stone to be cemented together were thoroughly saturated with water. In hot climates, like that of India, any work being built with cement should be kept wet and, if possible, under shelter during its progress, and for some time afterwards. Two bricks might be put together dry, as they came from the kiln, with the strongest cement that could be made, and if not previously soaked in water these bricks might be separated without the least difficulty, the joint being very little better than if made of dry sand. With bricks which absorbed from 1 lb. to 1 $\frac{1}{4}$ lb. of water, it was evident that, if left to absorb this moisture from the mortar with which they were cemented, they took away that which was necessary for crystallising or setting the cement."

The suggestion that the adhesive powers of mortar and cement are due to a process of crystallisation in contact with a roughened surface is strongly supported by the results of Mr. Grant's experience, as he lays the greatest stress upon the necessity for the presence of an ample supply of still water. The experience with the dry brick is most striking, as it is clear that such a brick would instantly absorb the moisture in the cement immediately in contact with it, and thus deprive it of the moisture necessary to bring about the process of crystallisation

CHAPTER VI.

ROUGH TECHNICAL TESTS OF MORTAR WITHOUT CHEMICAL ANALYSIS.

It is well known that in a large number of cases it is quite possible for a man having experience in certain work to detect almost at a glance the character of a material with



which he is daily working, and so it is with mortar. To the practical eye there can be no manner of doubt as to the kind of mixture which is being employed for the purpose of binding bricks. If it is good stuff, made of well-burnt lime and clean sand or grit, the fact is patent at once to the expert; and, so far as he is concerned, there is no need for a chemical analysis.

But when he has to satisfy others of the correctness of his opinion then it becomes necessary to resort to a method of registering in a definite and special way the facts on which his opinion is based, and this can only be done by having the sample in dispute properly analysed, and its component parts weighed on the balance, and the relative proportions of each material stated

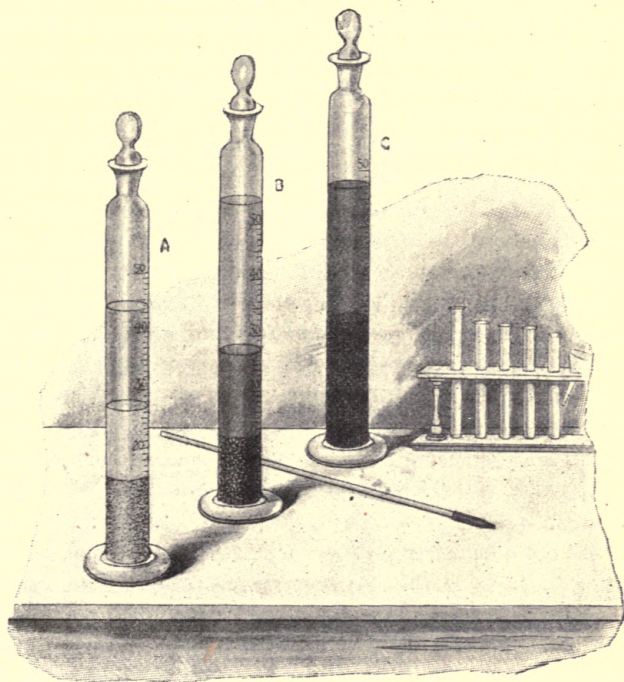
in precise figures. But if it is desired to place merely the general facts before an independent person to convince him that the mortar is not of a proper character, apart from the question of the actual quantities of the component materials, then this can often be done by the adoption of the following simple methods :—

(1) *Rough Test for Strength.*—Take a fragment of the mortar between the thumb and forefinger, and try to break it. If it crumbles easily into dust it is at once known that that its power of cohesion is slight, and that it will be wanting in binding power.

(2) *Test for Vegetable Débris, &c.*—Mix some of the mortar with an additional quantity of water, so as to make a thin paste with it, and then stir it well with a large quantity of water, adding the latter gradually, until the matters are thoroughly disintegrated. If road scrapings, &c., are present, light organic *débris* will be seen floating upon the surface of the water, and settling upon the top of the heavier mineral matters when the whole is allowed to rest.

(3) *Test for ascertaining the kind of Grit and relative proportions of Lime to Grit.*—For this test a series of small glass cylinders with feet, so that they will stand upright on a table, should be obtained. Their capacity should be about 100 cubic centimetres, or four ounces. Take about one ounce of the mortar and well mix it with about an equal quantity of water, then introduce it into one of the cylinders, and add more water until the vessel is nearly full. Now shake it thoroughly, having first closed the mouth of the cylinder with a cork, so as to well diffuse the ingredients of the mortar in the water, and thus separate the lime from the grit as far as possible. This will soon be accomplished, when the cylinder should be allowed to rest. The component parts of the mortar will now settle down in their

order of gravity—the grit first and then the earthy matter, and finally the lime. Of course, the line of demarcation between the respective substances will not be very sharp, but this will not matter for our present purpose. If the grit



ROUGH TECHNICAL TESTS.

is composed of good clean sand it will be seen at once. In the same way the presence of cinders, burnt ballast, &c., will be instantly detected. If the lime be well burnt and properly slaked, it will settle down last as a semi-flocculent

mass on the top of the grit; but if it be old mortar or imperfectly burnt lime, then it will settle down more after the manner of the grit itself, the difference between the behaviour of the two, viz., the good and bad lime, being most marked. If the student will try the experiment on half-a-dozen samples of mortar, some good and some bad, in as many cylinders placed side by side, he will gain such an insight into the method as can by no possibility be obtained from a mere written description.

(4) *Rough Test for Earthy Matter, &c.*—This test is very similar to the last, but with the addition of hydrochloric acid, known in commerce as “spirits of salt,” to the mixture of mortar and water. For this purpose it is better that the mixture should be made in a glass tumbler or china basin, as in the event of a bad mortar much frothing will take place on the addition of the acid. If the mortar is made with good, fresh, well-burnt lime very little effervescence will occur, the lime dissolving in the acid quietly; but if it is made with old mortar or badly burnt lime, then the acid will set free a large quantity of carbonic acid, which, if there be a material quantity of earthy matter or organic *débris* present, will cause a thick and dirty froth to rise often to the rim of a 10 oz. beaker, when even only a few grammes of the mortar are taken for the examination.

After the lime is all dissolved the earthy matters and grit will be left, and on the mixture being poured into one of the glass cylinders, and allowed to stand for a few minutes, it will be seen at once by mere inspection whether the grit is composed of proper or improper materials, such as road detritus, ash-bin refuse, garden soil, &c.

(5) *Tests for Setting Power.*—If a mortar is properly made it will set under water, but if not it will remain in a soft condition indefinitely. A convenient way to try this is

to place about 1 oz. of the mortar in question in one of the cylinders above referred to, and cover it with water. In this condition it should be allowed to rest for as long a time as may be convenient, say a week or more. After the lapse of sufficient time, if the mortar be pressed gently with a glass rod, it will, if of good quality, be found to resist the pressure, and be more or less hard; but if it be composed of rubbish, it will remain soft and allow the rod to be pressed right down to the bottom of the cylinder with ease.

These tests are in themselves absolutely sufficient to warrant the condemnation, or passing, as the case may be, of a given sample of mortar. But, unfortunately, they do not enable the exact proportions of the respective ingredients to be stated, and therefore, as I have said, chemical analysis must be resorted to for this purpose. They are sufficient, however, to enable one to show to independent persons, such as members of committees, &c., the general character of the substance which interested parties may be trying to persuade them is "good mortar."

It sometimes occurs, unfortunately, that there is a dispute as to the value of the indications of the results of an actual analysis. In such cases physical tests are of the utmost value. I was interested in a case recently in which I found 7 per cent. of "earthy matter," together with a large excess of carbonate of lime, and accordingly reported that the mortar was unfit for use. In consequence of representations made by the builder, I presume, a further sample was sent to another chemist, who evidently did not think that the quantity of earthy matter was of importance, as he reported in favour of the sample, with the result that the Local Authority took no action. I afterwards learnt from the surveyor who had charge of the case, that he had evidence that *earthy matter from under a hedge* was used in place of

clean grit. I think that there can be no doubt that if he had made such simple tests as are here recommended, and had placed them before his Committee, they would have supported the first analysis and conclusions, authorised action being taken to stop the use of the mortar, and obtained an order on the builder to pull down the defective brickwork.

CHAPTER VII.

THE CHEMICAL ANALYSIS OF LIME MORTAR, CEMENT, ETC.,
LIME AND LIMESTONE.

ALL samples should be first thoroughly broken up and mixed so as to ensure the small portion taken for the actual analysis being a fair representative sample of the bulk. The quantity so prepared should at once be placed in a wide-mouthed and well-stoppered bottle to preserve it from atmospheric influences, otherwise it will absorb carbonic acid, and possibly absorb or lose moisture, which would vitiate the results.

Lime and Limestone may be considered as synonymous, inasmuch as the lime is derived from the limestone by calcination, the difference being due to the carbonic acid which is driven out of the limestone in the process.

The chemical analysis of these substances may therefore be conducted upon identical lines, as the same constituents have to be looked for in each case. These are :—

Moisture.
Silica.
Oxide of Iron.
Alumina.
Lime.
Magnesia.
Alkalies.
Carbonic Acid.
Sulphuric Acid.

Several other substances are generally present, but these may be overlooked in a technical analysis.

For the estimation the following will be found to be a suitable method of procedure :—

- (1) 5 grammes are taken for moisture.
- (2) 5 grammes are taken for Silica, Iron, Alumina, Lime, and Magnesia.
- (3) 5 grammes are taken for Alkalies.
- (4) 1 to 2 grammes are taken for Carbonic Acid.
- (5) 5 grammes are taken for Sulphuric Acid.

(1) *Moisture*.—5 grammes of the sample are placed in a platinum dish and dried at steam heat, or preferably in an air bath at 103 deg. Cent. until it has a constant weight, the loss representing the moisture contained in the sample.

Objection has been taken to this method of ascertaining the quantity of moisture present in the sample, in so far as it may be assumed that carbonic acid will be absorbed by the lime during the process of drying; but a large number of experiments carried out by the author and Mr. R. Grimwood, have shown that such absorption, if any, makes so little difference that it is practically negligible.

(2) *Silica, Iron, &c.*—It is generally preferable, in the case of limestones especially, to place the 5 grammes taken for the analysis in a muffle furnace, and heat to bright redness for about an hour, by which means the carbonic acid is eliminated, together with any organic matter that may be present. By this precaution the excessive frothing which would otherwise take place when the sample is dissolved in acid is avoided, and consequently the loss from spurling.

The sample, when cool, is just moistened with strong hydrochloric acid, and allowed to stand for fifteen minutes, when practically the whole of the silica will be insoluble. Water then is added, and the whole passed on to a filter paper, and well washed till free from chlorides, and the filter paper, containing the silica, &c., dried, ignited thoroughly, and

weighed. When it is necessary to differentiate between sand and combined silica, it is necessary, after separating the heavy sand by elutriation, to heat the ignited silica in a platinum vessel on the water bath with pure hydrofluoric acid and a few drops of sulphuric acid, evaporate to dryness, ignite and weigh the residue. This may be repeated until a constant weight is obtained, the total loss by this treatment representing the combined silica.

Iron and Alumina.—The filtrate from the above is neutralised with ammonia, which is added in slight excess, raised to the boiling point, and set aside for the iron and alumina and settle out, or if the quantity be not large, they can be filtered from the liquid at once. Having been thoroughly washed with hot water, the precipitate is dried, ignited and weighed as the mixed oxides of iron and aluminium.

For the separation of these two constituents the weighed precipitate is re-dissolved in hydrochloric acid, and treated in either of the following ways :—

- a. Volumetrically by titration, with a standard solution of Bichromate of Potash, after reducing the iron to the ferrous condition by means of Stannous Chloride;
- or b. Gravimetrically by separating the alumina from the iron by means of a hot concentrated solution of caustic Potash.*

* The process is simple and reliable if the following directions are adhered to. Dissolve the weighed oxide of iron and alumina in hydrochloric acid. This is facilitated by carefully grinding the gritty ignited substance to a fine powder, placing it in a beaker, adding about 20 c.c. hydrochloric acid, placing the beaker on a wire gauze over a Bunsen burner, and covering the beaker with a large watch-glass, allowing the gas in the Bunsen burner to keep the acid simmering gently, until the iron and alumina dissolve. When this is

Lime.—The filtrate from the original precipitation of the mixed oxides of iron and aluminium is then treated with oxalate of ammonium, taking care that there is a good excess of ammonia present; boil briskly, remove the flame from the beaker, and add gradually about 2 grammes of fine-powdered pure solid ammonium oxalate while stirring continually. Again heat to boiling. On removing the flame, the precipitate will settle rapidly, and is easily filtered and washed. Having obtained the precipitate of oxalate of lime free from oxalate of ammonium, the lime may be estimated in several ways.

a. Volumetrically, by dissolving the precipitate in warm dilute sulphuric acid, and titrating with standard permanganate solution.

b. Gravimetrically, by the ignition of the dried precipitate

accomplished, about 50 c.c. of a strong solution of caustic potash should be placed in a beaker, and raised to boiling point over a Bunsen burner. The next part of the operation requires great care. If we add the whole of the hot acid, iron, and alumina solution at once to the hot caustic potash solution, there is a very great risk that the alumina will not be all re-dissolved. The proper method is to add the acid solution drop by drop to the potash, preferably letting it flow down a glass rod, which must be used after every addition to thoroughly stir the potash solution, and thus to ensure that the alumina, which is at first precipitated by the potash, is thoroughly re-dissolved before any further quantity of acid solution is added. From this it will be seen how necessary it is to have an excess of the potash over the quantity which would be necessary merely to neutralise the acid. When the whole of the acid solution has thus been added to the potash, the iron oxide will be in a state of suspension as a dark-brown woolly mass. The solution should now be boiled for a moment, and then filtered and well washed. As it is almost impossible to wash out all the caustic potash, it is better, after thoroughly washing, to re-dissolve the soft hydrated oxide of iron

in a muffle and weighing as lime (CaO); or by its solution in sulphuric acid, and precipitation therefrom as sulphate in the presence of alcohol.

In exact work the oxalate of lime first obtained should, after being thoroughly washed, be re-dissolved in HCl , and re-precipitated, as it invariably carries down small quantities of MgO .

The *Magnesia* is estimated in the filtrate from the lime precipitate by evaporating the liquid to dryness with a few drops of H_2SO_4 , and gently heating to drive off ammonium; dissolve in about 50 c.c. of distilled water; add an excess of ammonia and ammonium chloride, and then an excess of a solution of phosphate of soda; stir vigorously with a glass rod, and put aside to settle for twelve hours. An alternative method is to put the mixture of the solutions in a stoppered cylinder, and at once shake vigorously for five minutes, when

on the filter, with a few drops of hydrochloric acid, first taking care to place a clean beaker underneath the filter to collect the chloride of iron thus formed. When all the iron is dissolved, wash all the ferric-chloride from the filter paper into the beaker, add hot water, and then neutralise the acid with ammonia as in the first instance, after the removal of the soluble silica. This will precipitate the hydrated oxide of iron, which is now of course free from alumina. On filtering the chloride of potassium, formed by the action of the hydrochloric acid on the caustic potash retained on the filter and in the oxide of iron above described, it will be easily washed away, when the precipitated iron oxide can be dried, ignited, and weighed. This process is very much neglected in these days of volumetric work, but it is one which I have employed with great success. The whole secret is in the patience of the operator, who must be careful to add the acid solution very cautiously and slowly to the potash, which must be kept hot, and stirred well after each addition, and in re-precipitating the iron oxide with ammonia to get rid of the potash, which cannot be washed out directly.

the precipitation is completed without standing. Filter, dry, ignite, and weigh as magnesium pyro-phosphate, which, on multiplication by 0.36, will give the weight of magnesia (MgO).

(3) *Alkalis*.—The most accurate method for the determination of the alkalis, soda and potash, is that of Professor Lawrence Smith, which is fully described in Crookes' "Select Methods in Chemical Analysis." The limestone or lime is fused with one-quarter of its weight of ammonium chloride. When cool, the crucible with its contents are placed in a platinum dish, water added, and the whole slowly heated. By this means the fused mass becomes detached from the crucible and disintegrates. The crucible is then washed into the dish, and the liquid filtered. The filtrate from this contains the alkalis, together with some lime and magnesia. The liquid is heated, ammonia and ammonium carbonate are added in excess, and finally a few drops of ammonium oxalate. When settled, the liquid is filtered, and the filtrate and washings concentrated until ammonium chloride begins to crystallise out. The contents of the dish are then transferred to a flask, and strong nitric acid is added in sufficient quantity to decompose the ammonium salts present. Heat, and, when the reaction is ended, transfer to a porcelain dish, and evaporate to dryness. The residue is then boiled with baryta water (barium hydroxide) to remove magnesia, filtered, the excess of baryta being removed by ammonia and ammonium carbonate. The liquid from the last precipitation is filtered, and the filtrate evaporated to dryness with a few drops of hydrochloric acid in a platinum dish. The dish is carefully heated to ensure dryness, and when cold the mixed chlorides of the alkalis are quickly weighed.

An alternative method to the above is to dissolve the

lime or limestone in hydrochloric acid, make the liquid alkaline by the addition of an excess of milk of lime, and heat and filter. It need hardly be mentioned that the milk of lime here referred to must be made from freshly-ignited pure lime, slaked, and made into a cream with distilled water. By this means all metals, except those of the alkalis, are precipitated. The filtrate from this is then treated with ammonia, ammonium carbonate, and ammonium oxalate, in the same manner as described in the last method.

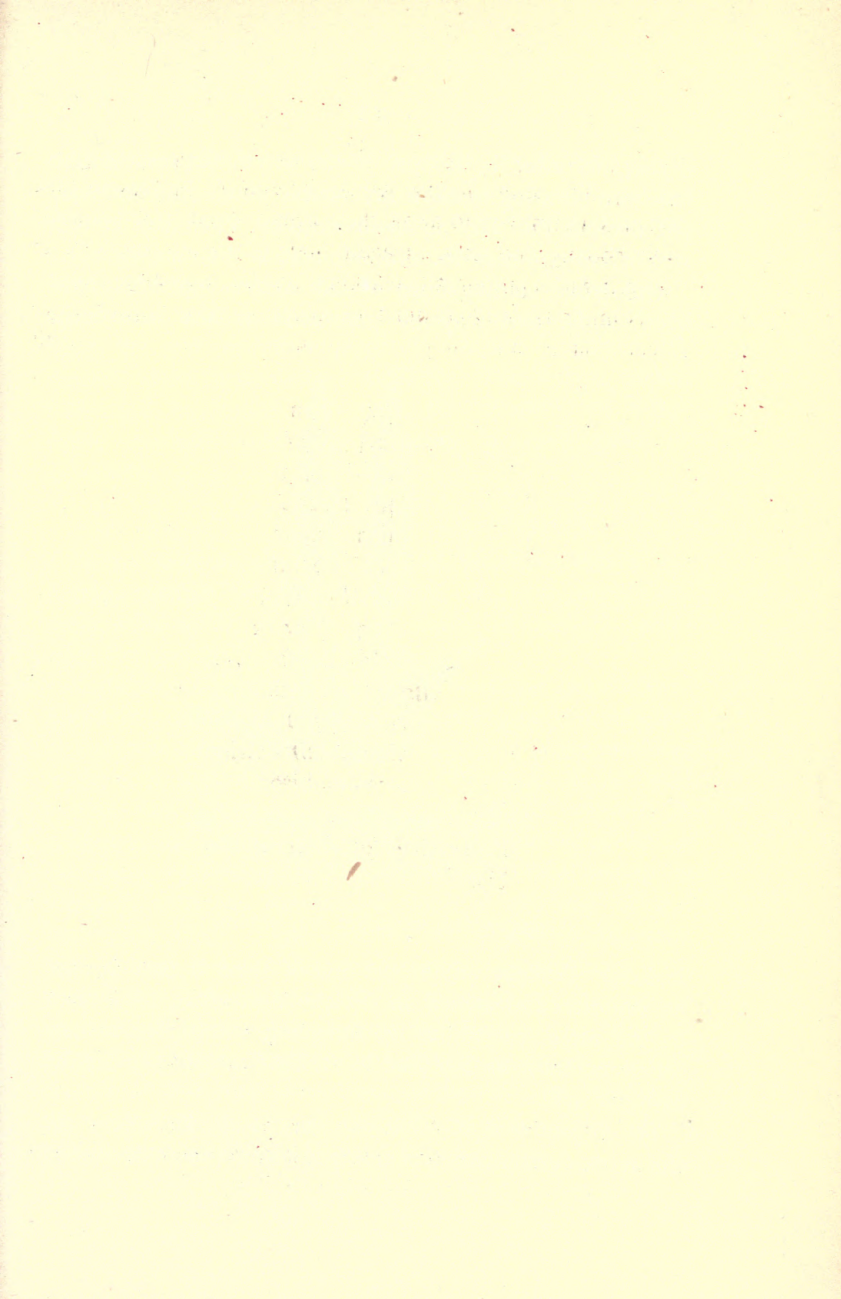
Separation of the Alkalis.—The weighed mixed alkalis are then dissolved in water, a few drops of hydrochloric acid added, and finally, an excess of platinic chloride solution. The dish is then heated on the water bath, until the contents are in a semi-fluid condition. A little alcohol is added, and the dish shaken to ensure mixture, when the double salt of potassio-platinic chloride crystallises out, and is filtered off, dried, and weighed. The weight so formed multiplied by 0.153, will give the quantity of potassium chloride, which, deducted from the mixed alkaline chlorides originally found, gives the quantity of sodium chloride.

Another method is to titrate the quantity of chlorine in the weighed mixed chlorides, and calculate the result from the following formulæ:—

$$\text{Weight of K} = \frac{[(A - B) 1.54] - B}{0.63}$$

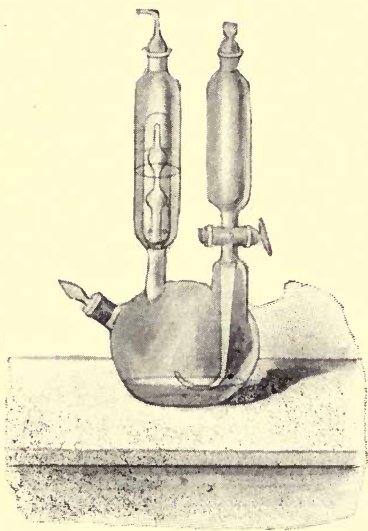
$$\text{Weight of Na} = \frac{B - [(A - B) 0.91]}{0.63}$$

Where A = the weight of the mixed chlorides, and B, the total weight of chlorine found in the chlorides, the weight of each alkali is usually calculated as oxide.



drying tube after the stopper on the end of the second tube has been removed to allow the access of air into the flask. On cooling, the flask is again weighed, when the loss in weight will represent the carbonic acid in the sample.

Numerous modifications of the above apparatus have been made, and may be purchased at the instrument



CHRÖTTER'S CARBONIC ACID APPARATUS.

makers. Perhaps one of the best of these is that devised by Schrötter and shown in the above woodcut. It is used in the same manner as the foregoing, except that concentrated sulphuric acid is used in the left-hand tube to dry the carbonic acid.

(5) *Sulphuric Acid*.—The contents of the flask used for the above determination of carbonic acid may now be washed

out and the sulphuric acid in it estimated as follows, viz.:—Evaporate to dryness, gently ignite, moisten with hydrochloric acid, and again dry, thus ensuring the complete insolubility of the silica. Moisten again with hydrochloric acid, add water, and filter, and to the clear filtrate add a solution of barium chloride; boil, and let stand to settle, when the precipitated sulphate of barium is filtered off, washed, dried, ignited, and weighed. The weight found, multiplied by 0.3435, will give the quantity of anhydrous sulphuric acid (SO_3) present in combination as sulphates.

MORTAR.

In the case of mortar, it is most important that a fair average sample should be obtained. For this purpose the preparation of the sample given under "Lime and Limestone" should be carefully carried out.

A portion of the powdered sample, weighing 20 grammes, is placed in a beaker, stirred up with a 10 per cent. solution of hydrochloric acid, and allowed to stand for twenty minutes in the cold, with frequent stirring. The fluid is then well stirred, and allowed to stand one minute, when it is decanted, and with it all the fine earthy matter, as well as the organic particles, held in suspension. This process is repeated until the supernatant water at the end of one minute is clear. The residue, which should be clean washed sand and grit, &c., is then dried by washing it into a platinum dish, draining off the water, drying the residue on the water bath, and weighing the dish with its contents from time to time until the weight remains constant. The weight of the dish being then deducted, the remainder represents the weight of "sand and grit" from the 10 grammes of the sample.



fine matter insoluble in the weak solution of hydrochloric is added to the sand and grit, and the above procedure adopted.

CLAY.

Clay being a constituent of cement, it is necessary to discuss this material in order to make the work complete.

As already pointed out in Chapter II., there are several varieties of clay, and for the purpose of analysis they may be divided into two classes, viz.:—

- (1) Alluvial Muds and Clays free from Lime.
- (2) Clay in which Carbonate of Lime is present.

In all cases it is desirable to work on the dried sample, and for this purpose the clay should be thoroughly dried in a water oven, when it should be at once ground into a powder and placed in a stoppered bottle

(1) *Alluvial Muds*.—Take 2 grammes of the dried sample and transfer to a platinum dish. Add about 25 c.c. of strong sulphuric acid, and dilute with 25 c.c. of distilled water. Gently heat for several hours, taking care not to raise it to the boiling point. Evaporate to dryness. When cold extract with hydrochloric acid, which is first added in sufficient quantity to just moisten the whole of the residue. This is then put aside to stand for fifteen minutes, when water is added, and the mass thoroughly disintegrated by means of stirring, &c., with a glass rod. The sand and silica are then filtered off, dried, and weighed. To separate the “combined” silica from the sand, the weighed sand and silica are repeatedly heated for some time (about 30 minutes) with successive portions of a strong solution of sodium carbonate. By this means the silica formerly in combination is dissolved, and the sand remaining behind is then washed, dried, and weighed, the difference between the

two weights giving the silica originally in combination with bases in the clay.

The hydrochloric acid solution is made up to a known volume, and a part of it examined for iron, alumina, lime, and magnesia; a part for alkalies; and another portion for the estimation of sulphuric acid, as described above.

(2) *Clay containing Carbonate of Lime*.—About 10 grammes of the clay are taken and treated with a slight excess of dilute hydrochloric acid, filtered through a weighed filter paper, washed, dried, and weighed, which will give the weight of clay present. In the filtrate, iron, alumina, lime, magnesia, &c., are estimated as under *Lime*. The clay is then treated as above described for *Alluvial Clay*. The whole of the results obtained are then calculated to percentages on the original sample.

PORTLAND CEMENT.

Two grammes of cement should be treated with a small quantity of water until all tendency to set has ceased. Strong hydrochloric acid is then added, and the whole evaporated to dryness and heated to about 150 deg. C. It is then moistened with strong hydrochloric acid, and again dried, to thoroughly decompose the silicates. It is again moistened with hydrochloric acid, water added, and the whole thoroughly stirred up and filtered, washed, dried, and weighed, the weight of the free silica *plus* sand, &c., being thus obtained. The free silica is then estimated as described under *Alluvial Muds* (see page 56).

The hydrochloric acid solution is now made up to a known quantity, and portions examined, as already described for iron, alumina, lime, magnesia, and sulphuric acid, under *Lime* (see page 42).

and W. Fresenius have worked out the following method of examination of Portland cement:—

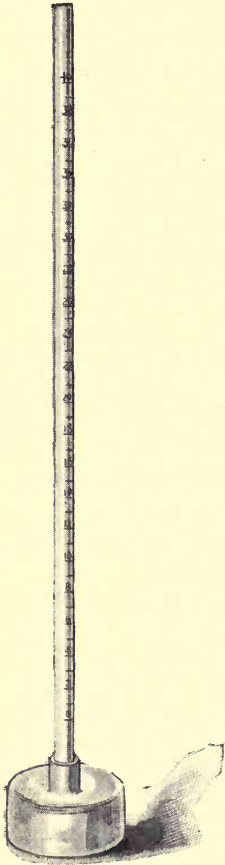
It is based on a comparison of some of the properties and behaviour under certain conditions of genuine cement, and of substances used for its adulteration, such as finely ground slag and hydraulic lime.

For instance :

- (a) Specific gravity.
- (b) Loss by ignition.
- (c) Behaviour to water ; that is, the alkalinity of the aqueous solution.
- (d) Behaviour to dilute acid.
- (e) Behaviour to Chameleon Solution (Permanganate of Potash).
- (f) Behaviour to Gaseous Carbonic Acid.

(a) The specific gravity is determined in the apparatus sketched on this page, and is based on an accurate determination of what *space* a *given weight* of the cement occupies.

In making a determination, the apparatus (see woodcut) is first filled to the zero point with turpentine ; 100 grammes of the cement is then introduced with the aid of a funnel into the apparatus through the graduated tube. The cement



FRESENIUS'
SPECIFIC GRAVITY
BOTTLE.

at once falls to the bottom, and the liquid rises in the tube. The number of c.c. read off on the tube represents the volume occupied by the 100 grammes of cement. By dividing the volume in c.c. into the weight, the specific gravity is obtained.

Precautions necessary in Manipulating.

Keep the lower portion of the apparatus in water at the air temperature, and control by a thermometer, as constancy of temperature is very important.

Give the apparatus a few gentle taps in order to facilitate the escape of air bubbles.

Close the tube with a cork until complete subsidence has taken place, in order to prevent evaporation of the liquid. The results of two experiments ought not to differ more than one unit in the second place of decimals.

The sample of cement in the above and for the following experiments should be so finely pulverised as to pass through a sieve with 5000 meshes to the square centimetre; equal to 32,257 per square inch, or about 180 per lineal inch.

(b) *Loss by ignition* is determined by heating 2 grammes over an ordinary Bunsen lamp, with chimney, until weight remains constant.

(c) *Behaviour to Water* is determined by agitating 1 gramme of finely powdered cement with 100 c.c. distilled water at ordinary temperature for ten minutes. After filtration, 50 c.c. are titrated with $\frac{N}{10}$ H Cl.

(d) *Behaviour to Diluted Acid* is determined by agitating 1 gramme of substance, finely powdered, for ten minutes with a mixture of 30 c.c. normal acid and 70 c.c. water. After filtration through a dry filter, 50 c.c. are titrated back with normal soda solution, and from this the amount of

0.5 gm. substance should correspond to 4 to 6.25 c.c. $\frac{N}{10}$ acid.

- (d) The consumption of between 18.8 c.c. and 21.67 c.c. of normal acid by 1 gm. of cement.
- (e) The consumption of between 0.79 and 2.80 m.g. of chameleon solution by 1 gm. cement.
- (f) An absorption of from 0 to 1.8 m.g. C O₂ by 3 gm. cement.

Cements giving results which do not come within the limits given must be looked upon either with suspicion, or as beyond doubt adulterated, according to the results.

Table of Results of Experiments on Adulterated Cement.

Description and composition of the mixture.	Specific gravity.	Loss by ignitor, per cent.	Alkalinity of aqueous solution, $\frac{1}{10}$ N & A, corresponding to 0.5 gm. cement.	1 gm. requires c.c. N acid.	1 gm. reduces m.g. of permanganate.	3 grms. absorb m.g. C O ₂
	(a)	(b)	(c)	(d)	(e)	(f)
(1) One part hydraulic lime, nine parts Portland cement	3.067	1.90	6.50	20.50	not determined	4.6
(2) One part hydraulic lime, nine parts Portland cement	3.053	2.52	8.20	20.04	not determined	3.6
(3) One part of slag meal, nine parts of Portland cement	3.114	2.04	3.8	19.53	6.11	1.6
(4) One part of pulverised slag, nine parts of Portland cement ...	3.115	1.59	4.0	20.6	8.31	0.7

The results under the headings *a, c, d, &c.*, apply to adulteration with slag ; those under the headings *a, b, c,* and *f* are of use in the detection of adulteration with hydraulic lime.

Interpretation of Table on page 64.

No. 1 shows too low specific gravity. Loss by ignition is not normal. Alkalinity of aqueous solution too high, and too great an absorption of CO_2 .

No. 2 shows too low specific gravity, a very high loss by ignition, a high alkalinity of the aqueous solution, and too great an absorption of CO_2 . In both cases *sufficient proof* of adulteration by hydraulic lime.

Nos. 3 and 4. An admixture of slag meal can be recognised by the large amount of cameleon solution used. In No. 3 the alkalinity of the aqueous solution is below the limit, while in No. 4 it is just reached. The specific gravity of both is below that of genuine Portland cement, viz. 3.125. The consumption of acid gives no information.

On page 63 is a useful form of table for recording the results of the chemical and physical examination of a sample of cement.

CHAPTER VIII.

TYPICAL ANALYSES OF MORTAR AND CEMENT.

DURING the past twenty years I have had opportunities of analysing various samples of cement and mortar, and of the sand which has been adopted as a standard for use in testing the strength of cement—having been associated with the late Mr. John Grant, engineer in charge of the southern district of London and the building of the southern portion of main drainage system under the late Metropolitan Board of Works, whose work on cement has for years been the standard of reference in regard to the mechanical tests, &c. From my results obtained in the course of this work, I have selected the following series of typical analyses, and have added thereto the results of the analyses of mortar, &c., which were first published in a paper read before the Society of Public Analysts by myself and Mr. R. Grimwood, F.I.C., &c., in 1896. As the value of this paper is greatly enhanced by the discussion thereon, I venture to print the whole *in extenso*, except that portion devoted to analytical methods, which have been already fully dealt with. Together these analyses form a sequence which will be of interest and value to those who are desirous of ascertaining the character of samples which have been submitted to analysis, as by comparing the results found with the following they will be able to assign to any sample its proper position in the scale of quality. Thus, it may be a mixture of good materials, or of adulterated materials, or one which, though honestly made up by the workman, has yet been mixed with poor mate-

rials. I do not intend to imply, however, that the mere analysis of a particular sample will indicate the setting power of a cement. This, it will be seen on consideration, must be affected more or less, if not entirely, by the physical character of the cement, especially in regard to the manner of its burning, grinding, &c. A cement may contain all the ingredients of a first-class cement, but if it is not properly prepared it will be useless.

THE LEGAL SPECIFICATION OF MORTAR.

In the bye-laws made by the London County Council, under Section 16 of the Metropolitan Management and Building Acts Amendment Act, 1878, it is provided that the mortar used under that Act must be composed of freshly-burnt lime and clean, sharp sand or grit, without earthy matter, in the proportions of one of lime to three of sand or grit.

In the case of cement-mortar, the cement to be used must be Portland cement, or other cement of equal quality to be approved by the District Surveyor, mixed with clean, sharp sand or grit, in the proportions of one of cement to four of sand or grit; also that burnt ballast or broken brick may be substituted for sand or grit, provided such material be properly mixed with lime in a mortar mill.

In the bye-laws made by the Council under Section 31 of the London County Council (General Powers) Act, 1890, it is provided that plastering or coarse stuff shall be composed of lime and sand, in the proportion of one of lime to three of sand, mixed with water and hair; but Portland, Keene's, Parian, Selenitic, or other cement or plaster of Paris, may also be used for plastering. The lime must be freshly-burnt lime; the sand must be clean, sharp sand, free from loam or earthy matter; the hair must be good

and sound, free from grease or dirt, and one pound of hair be used to every three cubic feet of coarse stuff. Fibrous material may be used instead of hair, and ground brick or furnace slag, each to the satisfaction of the District Surveyor, may be used instead of sand; and the setting coat must be composed of lime or cement mixed with clean washed sand, or cement only.

USUAL CHARACTER OF MORTARS.

Earthy Matter.—These extracts from the bye-laws relating to the use of building materials in the metropolis, stating the exact proportions, enable an analysis of lime or cement mortar, &c., to indicate very slight departures from the prescribed quantities of the materials used. Unfortunately, analyses which have been made from time to time by us (Dibdin and Grimwood) have shown distinctly that it is only under exceptional circumstances that mortars which come within these regulations are employed. To take one factor only, the earthy matter. Analyses of samples of mortar, made with materials selected as coming within the definition of the bye-laws, and in the proportions therein set out, show that the earthy matter, as determined by us in the manner already described, varied as follows:—1·5, 2·1, 0·14, 0·74, 0·81, 0·61, 0·64, 0·96, 0·85, and 0·70 per cent.; thus showing that the finely-divided matters which may be accidentally introduced by means of the broken brick or sand, form but a very small proportion of the actual weight of the mortar. As compared with these, the following results obtained from the examination of a number of samples of mortars taken from buildings in course of erection will indicate the great difference in this



respect, viz. : 4·9, 3·8, 9·1, 3·4, 14·7, 13·7, 7·4, 10·4, 8·1, 12·3 per cent.

Soluble Silica.—Another factor of importance in estimating approximately the quantity of cement in a genuine cement mortar is the soluble silica. As is well known, the silica present in Portland cement, soluble in hydrochloric acid, will vary between 17 and 20 per cent.; but if the average is taken at about 18 per cent., it will form a fair basis for calculation.

If the cement mortar is made with good Portland cement, and clean well-burnt brick or sand, in the proportion of one of cement to four of the grit, there will be about 3·5 per cent. of soluble silica. In some samples of genuine, but badly mixed, cement mortar, the soluble silica was found to vary from 2·0 to 3·25 per cent.; and some samples of genuine mortar made with lime gave soluble silica equal to 0·9, 1·0, 0·6, traces, and 1·1. If we now turn to the analyses of commercial mortars (see Table V.) above referred to, we find that the soluble silica varied as follows: 0·9, 0·7, 0·7; thus showing a marked difference in this factor in good and bad mortars, &c.

Carbonic Acid.—A third factor is the quantity of carbonic acid. Genuine mortars made with good materials were found to contain, even after the mortar had been made for some three months, only carbonic acid equal to 1·02, 1·36, and 1·06 per cent. of carbonate of lime; and some cement-mortars 2·6, 2·86, 1·04, 1·87, 1·45 per cent. of carbonate of lime. Freshly-prepared mortar made with good materials contained in two instances 0·3 and 0·4 per cent. carbonate of lime. Against these, some commercial mortars contained 6·42, 6·24, 7·39, 4·52, 8·9, 8·4, 8·0, and 4·5 per cent.; thus showing conclusively, that this factor alone gave such marked indications of the character

of the material that it would be almost impossible that a mistake could be made in estimating the character of a given sample of mortar or cement. It should be noted that certain bricks contain carbonate of lime, and this should be determined before concluding that the mortar is made with old materials or insufficiently burnt limestone. In Table I. an instance is given of a brick containing 7.77 per cent. of carbonate of lime.

TABLE I.

Sample of Lime used in making Mortar.

	Per cent.
Lime (CaO)	78.40
„ carbonate	3.22
„ sulphate	0.64
Soluble silica, iron oxide, and alumina.. ..	12.85
Sand	4.80
	<hr/>
Total	99.91

Sample of Brick used in making Mortar.

	Per cent.
Moisture (loss at 212 deg. Fah.)	1.27
Lime (CaO)	1.99
„ carbonate	7.77
„ sulphate	1.03
Soluble silica, iron oxide, and alumina.. ..	3.60
Insoluble matter—coarse	78.77
Insoluble matter—fine (this had the appearance of having been burnt)	1.69
	<hr/>
Total	96.12

*Relation of Weight to Volume (Lime Mortar).—*In the bye-laws of the London County Council it is provided that the quantities are to be by volume, and not by weight; therefore the analysis, being made by weight, must be converted into terms of volume. In order to show how this is

arrived at, the following comparative analysis of a sample of mortar made with broken brick, specially prepared for this purpose, will best make the point clear. The analyses of the lime and broken brick are given in Table I., and in Table II. that of the mortar made with one volume of lime and three volumes of brick. In this case the brick had some old mortar adhering to it, as would happen where old bricks are used.

TABLE II.

Sample of Mortar made with One Part by Measure of Lime and Three Parts Crushed Brick.

	Per cent.
Moisture, water of hydration, &c.	29.71
Lime (CaO)	12.85
(= 16.06 commercial lime.)	
Lime, carbonate	4.47
„ sulphate	0.61
Crushed brick	46.03
Iron oxide and alumina	2.80
Soluble silica	1.85
Earthy matter (this had the appearance of having been burnt)	0.70
Loss on ignition	0.98
Total	100.00

Commercial lime (containing 80 per cent. of CaO) to crushed brick, 1 to 2.29 by volume; to all other matters, 1 to 2.86 by volume (see page 3).

In this it will be seen that the earthy matter was 0.70 per cent. in the mortar, and the lime (as pure lime) 12.85 per cent., equal to 16.06 per cent. commercial lime of 80 per cent. pure CaO.

The brick (Table I.), as already mentioned, contained 7.77 per cent. of carbonate of lime. Another instance is given in Table III. of the analyses of lime and good brick, and mortar made therewith. In this the carbonate of lime was only 1.97 per cent. on the mortar.

TABLE III

Analyses of Stone-lime and Mortar made therefrom

	Stone lime.	Mortar. Lime 1: brick 3 parts by volume.
Moisture, water of hydration, and loss	1·86	.. 31·23
Lime (CaO)	78·63	.. 10·89
Lime carbonate	0·93	.. 1·97
Lime sulphate	None	.. 0·61
Iron oxide and alumina	4·30	.. 3·55
Soluble silica	7·10	.. 1·11
Sand and grit	Trace	.. 45·99
Earthy matter	6·20	.. 1·37
Loss on ignition	0·98	.. 3·28
	100·00	100·00

A large number of experiments on various samples of lime have shown that it is a safe rule to increase the weight of lime found in the analysis by one-fourth in order to raise the lime (CaO) obtained to the relative quantity of *commercial* lime, on the assumption that the commercial lime originally contained 80 per cent. of pure oxide of calcium. The correction from weight to volume is then made by means of the relative specific gravities of the lime and sand or grit.

The following will illustrate the method:—

Equal volumes of stone-lime and of air-dry broken brick weighed as follows:—

Lime	17·38	grammes	per	18·5	c.c.
Brick	21·13	"	"	"	"

(1) *To convert the weight of commercial lime to a volume corresponding to that of broken brick, add one quarter of the weight of lime found, to itself, thus: 17·38 + 4·34 = 21·72, as compared with 21·13 of brick.*

(2) By analysis this lime contained 78.63 per cent. of real CaO, and a mortar made with this lime on analysis gave 10.89 per cent. of CaO; therefore, to convert CaO in the mortar into terms of commercial lime, for practical purposes add to the weight of CaO found one quarter of itself. Thus, CaO in mortar = 10.89 per cent. + 2.72 = 13.61 commercial lime.

(3) By No. 1, $13.61 + 3.4 = 17.0$ of CaO by volume.

(4) The ground brick used in making the mortar (Table III.) contained 6 per cent. of matters soluble in weak acid; therefore, to the weight of brick-grit actually found add 6 per cent., *i.e.*, sand and grit (brick), $45.99(45.99 \times 0.06 = 2.76) = 48.75$.

(5) Lime to brick by volume = 17 : 49, or 1 : 3 nearly.

The following table of the weight of various sands and other substances per cubic foot will be of assistance to the reader. This table should not, however, be relied upon in the case where a sample of the material under consideration is obtainable; in such a case it should be weighed under normal working conditions. For comparison, I have also given the weight of various limes, but in this case also it will be best, wherever possible, to make a direct determination in each case. The weighings under the head of "Sand, &c.," have been specially made for the purpose of this chapter by Mr. C. Chambers Smith, C.E.

WEIGHT OF ONE CUBIC FOOT OF SAND, &c.

	lb.		lb.
Pi sand.. .. .	100	Burnt clay	71
River sand	118	Cinders, clean	60
Broken brick	53	Clinker	50

WEIGHT OF ONE CUBIC FOOT OF FRESH LIME.

In small lumps.	lb.	Ground.	lb.
White chalk	36	White chalk	54
Grey chalk, Halling*..	40	Grey chalk, Halling*..	58
Portland stone	47	Arden	68
Blue lias	38 to 50	Blue lias	49 to 68

* Flare-burnt grey lime.

CEMENTS.

	lb.		lb.
Portland.. .. .	74 to 101	Parian	60
Roman	60 to 62½	Plaster of Paris	50
Medina	61	Whitening	64
Keene's	64		

RELATION OF WEIGHT TO VOLUME (CEMENT - MORTAR.)

Equal volumes of washed and dried sand and of Portland cement of specific gravity 3.15 weighed 21.7 grammes and 19.3 grammes respectively. Therefore the weight of cement found must be raised by one-eighth to bring the materials into equal terms of volume.

As already stated, average Portland cement will contain from 17 to 20 per cent. of soluble silica. An average of 18 per cent. may therefore be taken, on which assumption the soluble silica formed may be calculated into terms of cement. Thus, the 3.25 per cent. of soluble silica found in sample No. 1, Table IV., would equal 18 per cent. of cement, or 19.25 per cent. corrected for volume. The sand and grit equalled 67.34 per cent.; therefore the ratio of cement to sand was 1 : 3.5. In sample No. 2 of the same table the soluble silica was 2.5 = 13.9 per cent. of cement = 15.6 by volume; the sand, &c., was 70.77 per cent.; the ratio consequently was 1 : 4.5. Sample No. 3 showed a ratio of 1 : 3.5, No. 4 of 1 : 5.6, and No. 5 of 1 : 4.8, the average

being 1 : 4.4. The samples were stated to be from cement mortars, made with 1 of cement to 4 of sand, doubtless the differences are due to unequal mixing.

ABSORPTION OF CO₂ AFTER MORTAR IS MADE.

The crushed brick (see Table I.), including some of the silica, iron oxide, and alumina, would amount to 48 per cent., or 51.77 per cent., including 3.74 per cent. of carbonate of lime found on analysis to be present in the brick. The commercial lime used contained 3.22 per cent. of carbonate of lime, so that the above 16.06 per cent. would contain 0.52 per cent. These together will account for 4.26 per cent. of the carbonate of lime in the quantity found in the above analysis, showing that only 0.21 per cent. of carbonate of lime was formed in the time between the making of the mortar and its analysis.

The appended tables, Nos. IV. to VII., contain the results of the analyses of some typical samples.

Table No. IV. shows the results of the analyses of five samples of cement mortar.

Table No. V. shows the results of the analyses of three samples of bad mortar.

Table No. VI. shows the results of partial analyses of fifteen samples of very bad mortar. In these the analyses were carried only far enough to show that all the samples contain excessive quantities of earthy matter and old mortar, and also that they were deficient in freshly-burnt lime.

In contrast with these, Table No. VII. shows the result of the partial analysis of a sample of good mortar taken from a public building in course of erection. In all the following tables the results are stated in percentages.

TABLE IV.—*Analyses of Samples of Cement Mortar*

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Moisture, water of hydration, &c.	7.50	6.12	7.72	7.26	9.68
Lime (CaO)	11.80	10.87	11.97	9.20	11.24
„ carbonate	2.86	2.86	1.04	1.87	1.45
„ sulphate	0.20	0.30	0.99	0.84	0.71
Iron oxide and alumina	4.35	3.75	4.00	4.95	4.00
Soluble silica	3.25	2.50	3.25	2.00	2.25
Earthy matter	0.61	0.64	0.96	0.85	0.70
Loss on ignition	2.09	2.19	2.42	2.50	2.02
Sand and grit	67.34	70.77	67.65	70.53	67.95
Total	100.00	100.00	100.00	100.00	100.00

Ratio of Cement to Sand, &c.

Calculated on the Soluble Silica.

No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
1:3.5 ..	1:4.5 ..	1:3.5 ..	1:5.6 ..	1:4.8

Calculated on the Lime.

1:2.7 ..	1:3.0 ..	1:2.9 ..	1:3.0 ..	1:3.0
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TABLE V.—*Bad Lime Mortars.*

	No. 1.	No. 2.	No. 3.
Moisture, water of hydration, &c.	17.1	21.1	15.1
Lime (CaO)	5.9	6.0	6.2
„ carbonate	8.9	8.4	8.0
„ sulphate	1.8	1.7	2.0
Sand, grit, broken brick, &c.	51.5	46.3	53.4
Iron oxide and alumina	1.6	1.4	1.5
Soluble silica	0.9	0.7	0.7
Earthy matter	7.4	10.4	8.1
Loss on ignition	4.9	4.0	5.0
Total	100.0	100.0	100.0
Commercial lime (CaO) to sand and grit, &c., by volume	1 to 5.5	1 to 4.9	1 to 5.5
Commercial lime (CaO) to all other matters by volume	1 to 8.3	1 to 7.7	1 to 8.1

TABLE VI.—*Partial Analyses of Very Inferior Mortars.*

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.	No. 7.
Moisture	3.13	3.61	3.42	2.80	2.79	3.00	3.22
Lime (CaO)	7.48	8.72	5.68	7.71	6.11	6.00	6.73
Lime carbonate	12.61	13.18	15.45	11.36	15.79	14.66	16.34
Sand, grit, &c.	58.90	54.85	57.14	58.76	59.09	55.36	55.84
Earthy matter	8.44	7.39	8.68	7.57	7.65	9.94	9.27
Iron oxide, alumina, and soluble silica	3.75	2.90	3.00	4.50	3.30	2.00	3.20
Commercial lime (CaO) of 80 per cent. to all other matters by volume	1 to 5.0	1 to 4.2	1 to 6.4	1 to 4.8	1 to 6.1	1 to 5.9	1 to 5.3
Commercial lime (CaO) to all other matters by volume	1 to 7.6	1 to 6.1	1 to 10.2	1 to 7.4	1 to 9.5	1 to 9.7	1 to 8.5

	No. 8.	No. 9.	No. 10.	No. 11.	No. 12.	No. 13.	No. 14.	No. 15.
Moisture	3.87	10.28	4.64	3.68	3.93	7.97	3.15	6.32
Lime (CaO)	9.97	9.00	7.50	12.01	7.63	10.29	8.34	5.96
Lime carbonate	11.25	11.13	15.11	8.86	15.80	8.63	16.70	20.95
Sand, grit, &c.	57.00	50.96	50.35	55.27	50.60	54.27	50.35	52.05
Earthy matter	8.10	8.78	9.30	8.63	8.78	8.70	10.41	8.23
Iron oxide, alumina, and soluble silica	4.50	3.60	3.80	4.15	4.70	3.70	4.20	3.30
Commercial lime (CaO) of 80 per cent. to sand, grit, broken brick, &c., by volume	1 to 3.6	1 to 3.6	1 to 3.9	1 to 2.9	1 to 4.1	1 to 3.3	1 to 3.8	1 to 5.5
Commercial lime (CaO) of 80 per cent. to all other matters, by volume	1 to 5.5	1 to 5.6	1 to 6.9	1 to 4.4	1 to 7.2	1 to 5.0	1 to 6.7	1 to 9.4

The average total lime in cement may be taken as 60 per cent.; therefore, the lime found, including that carbonated, may be corrected to volume of cement thus:—Sample No. 1—CaO = $11.8 + 1.6$ (which had become carbonated) = $13.4 = 22.3$ of cement; add one-eighth for correction to volume = 25.1 vols. of cement to 67.34 of sand, or a ratio of $1 : 2.7$.

TABLE VII.—*Sample of Good Mortar taken from a Public Building in Course of Erection.*

Moisture	17.84
Lime (CaO)	14.43
Lime carbonate	0.97
Sand, grit, &c.	59.15
Earthy matter	1.05
Iron oxide, alumina, and soluble silica ..	3.75

Commercial lime (of 80 per cent. CaO) to sand, grit, &c., by volume, 1 to 2.6.

Commercial lime (of 80 per cent. CaO) to all other matters (dry) by volume, 1 to 3.0.

It will be noticed, however, that the calculation of cement from the lime found is unsafe, as the mortar might be sophisticated with lime, and therefore not *cement* mortar. The soluble silica is the only reliable indicator.

In opening the discussion which followed the reading of the paper, from which the above is extracted, —

Sir Charles A. Cameron said he was afraid he could not add anything to the very valuable information that had been laid before the Society by the authors of this paper. He occasionally had specimens of cement submitted to him which, although apparently having a correct composition—according to what was laid down in the text-books—nevertheless would not adhere to the surface of bricks. As a matter of fact, disputes were constantly arising in connection with cement, mortar, and concrete, and there was a very large field open to workers in the direction of obtain-

ing information which would enable such disputes to be settled on a scientific basis. In Ireland he did not think it was customary to use ground bricks in place of sand to such an extent as appeared to be the case in England. He believed the authors had mentioned the case of a mortar in which the proportion of lime to sand was as low as 1 : 9. He had himself once examined a sample containing almost as much sand, viz., 86 per cent.

Mr. Allen said that one problem which he had been called upon to solve was how much road-sweepings a particular sample of mortar contained, and what was the proportion of "clean, sharp sand." Road-sweepings seemed to be a very indefinite article, and he thought that if the authors could indicate how to ascertain the quantity present, it would be of great service to others as well as to himself. It seemed to him, if he had understood that part of the paper correctly, that it was a little questionable whether it was fair to deduce the amount of cement in a cement mortar from the amount of soluble silica. This surely must be a very variable quantity. There was no doubt that the Society was very much indebted to anyone bringing forward figures on such a difficult subject, as they would at least afford something like a basis for further investigations.

Mr. Bodmer remarked that where broken bricks were used in substitution for sand, the bye-laws, although providing for the proper grinding up of the bricks, did not specify distinctly that they should be well burnt. Badly-burnt bricks might be used, and in that case the amount of earthy matter present would be considerable. He thought it desirable that either the use of bricks should be altogether prohibited, or that some more definite limit should be laid down as to their employment.

The proportion of quicklime contained in the lime used

was also a matter upon which disputes often arose, but it was hardly possible to suggest any definite standard, for in some kinds of lime, which were really very good for mortar making, the percentage of actual quicklime was comparatively low.

Mr. John Hughes said that the bye-laws of the London County Council in regard to the composition of mortar were far too vague. Nothing is mentioned respecting the quality of the lime, except that it should be "freshly burnt," its richness in CaO and soluble silica is entirely omitted, as well as its weight per cubic foot, which may vary considerably. The sand must be sharp and clean, "without earthy matter," but there is no definition of what constitutes earthy matter.

In making mortar it is usual to specify quantities by measure rather than by weight, consequently the respective amounts of lime and sand, or substitutes for sand, must vary in proportion to their gravity. Therefore mortar, though made according to the strict reading of the bye-laws, may contain as little as 10 parts of lime (CaO) by weight in every 100 parts of the perfectly dry mortar. Thus, taking a cubic foot of grey lime containing 80 per cent. CaO, and weighing (according to Hurst's "Architectural Hand-book") 44 lb., and three cubic feet of clean sand weighing 100 lb. per cubic foot, we have a mixture in the dry state which contains only 10.23 parts of CaO in every 100 parts by weight. On the other hand, if a substitute for sand, such as broken brick, having a less gravity, be used, the percentage of CaO in the mortar would be increased. Some years ago he devoted considerable time to the examination of old mortar obtained from numerous old abbeys and castles, and the results of the analyses were published in *The Builder* during the years 1892 and 1893. These analyses corroborated

Mr. Bodmer's observation, that the percentage of actual quicklime was really no criterion of the quality of the mortar. The lime rapidly becomes converted into carbonate, especially in the smoky atmosphere of towns, and in the case of good building lime the CaO is associated with soluble silica in the same form as it exists in Portland cement, so that the total lime in its various combinations, rather than the caustic lime alone, should be considered before condemning a mortar.

He would mention that, according to his researches, the average amount of caustic lime in ancient mortar did not exceed 0.5 per cent. Further, within certain limits the actual percentage of total lime was no reliable indication of the quality of the mortar. Thus the mortar from Rochester Castle (of which only the keep remains) contains as much as 28.67 CaO, while that from Tintern Abbey, in Monmouthshire (which was remarkable from the fact that the four gable ends were still standing), contains only 18.84 CaO per cent. The mortar from the leaning tower of Caerphilly Castle, in Glamorganshire, contains as little as 13.49, associated, however, with 9.85 of soluble silica. He could not agree with Mr. Dibdin in calculating the probable amount of cement present in a mortar from the figures for soluble silica. Limestones contain varying quantities of gelatinous or hydrated silica, which after calcination forms a natural cement, and it is to the presence of this cement that the durability of ancient mortar largely depends. Greystone lime contains 9 per cent. of this soluble silica; Aberthaw lime contains as much as 15, while the best Portland cement contains not more than from 20 to 22; consequently limestones are suitable for making mortar in proportion to their richness in this so-called soluble silica. In conclusion, he would say that some of the best and most

durable of these old mortars contained as much as 4 to 5 per cent. of oxide of iron and alumina.

Mr. B. E. R. Newlands said he presumed this paper was intended to refer only to cases occurring in those districts in which the bye-laws referred to had been adopted; but there were districts where the bye-laws had not been adopted; and a still greater number where they had not been enforced. In Manchester, and other large towns, mortar was manufactured as an industry by the Corporation, and the mortar was generally made with the cinders and clinkers from the Corporation's destructors. This mortar—which was sold to the general public by the Authorities—would yield very different analytical results from the mortars dealt with in this paper. Mortar made with furnace clinker was also used to a considerable extent in the metropolitan area—most frequently, perhaps, in the case of buildings erected by works proprietors whose furnaces supplied the clinker; and this mortar, although of a very high standard as far as hardness and setting power were concerned, would not satisfy the tests given in the paper.

Dr. M. A. Adams said that, from the local experience he had gained in the midst of the cement district, where there was an abundance of materials for making lime from both white and grey chalk, he could confirm the remark made by Mr. Hughes, that alumina was often a somewhat important constituent of commercial lime. It was customary in his neighbourhood to look upon the lime made from grey chalk (which contained a large proportion of silicates and 3 or 4 per cent. of alumina) as almost equal in quality to Portland cement. Certainly, it was a fact well known to all practical builders in the district, that they could use far less of such lime than was required if they employed lime made from

the upper layers of the chalk, which was nearly pure calcium carbonate.

The author, in replying to the discussion, said that he and his colleague congratulated themselves on having raised such an interesting discussion. He sincerely hoped that the paper would be the forerunner of several others on the same and kindred subjects. Mr. Newlands was perfectly correct in his remarks as to the scope of the paper. The question had been dealt with entirely as under the bye-laws relating to London. As a matter of fact, these bye-laws contained the only statement or definition of mortar having legal weight of which he was aware. If they were wrong, it was without doubt desirable that they should be amended as soon as possible, but as they existed, a considerable amount of work had been done under them, and it was just as well that they should be thoroughly understood. The subject, taken in its industrial aspect and as ranging over the ground of all kinds of mortar, both ancient and modern, was of course a very large one, and to treat it exhaustively would require a great deal more than the hour or so available at one of the Society's meetings, and he and his colleague looked upon this paper rather as an index for pointing out that a large amount of valuable work could be done by those who took up the subject in detail. With regard to the adulteration of mortar, it was seldom that a sample was found which really complied with the specification contained in the bye-laws, a common adulterant, in London at any rate, being dust-bin refuse. Mr. Allen had asked for information as to the best mode of estimating road-sweepings. In many cases road-sweepings consisted largely of sharp sand mixed with organic matter. The only way to deal with these was to separate the organic from the mineral matter, and estimate them in the ordinary way. Of course, quite a cursory observation would indicate

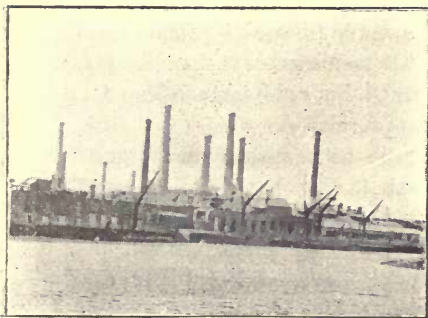
whether the organic matter was in the form of horse-dung or the like. With regard to Mr. Bodmer's remarks, he did not think much difficulty would be found in differentiating between properly-burnt earthy matter and clay that had merely been warmed up. The bye-laws appeared to be perfectly definite in their references to the ballast or bricks that might be used in making mortar. They stated that burnt ballast or broken brick might be substituted for sand or grit. Now, earthy matter that had merely been warmed could not properly be described as "burnt ballast," and unless a brick has been fired—and properly fired—it could not, according to his (Mr. Dibdin's) idea, be regarded as coming within the meaning of the term "brick," any more than partially heated chalk could be regarded as lime. Mr. Hughes had remarked that the amount of lime present was no criterion of the quality of a mortar, and had referred to the mortar of Tintern Abbey as containing 18.84 per cent. of lime. Now, assuming 18.84 per cent. of caustic lime to be equivalent to 23.55 per cent. of commercial lime, and adding one-fourth to correct for the difference in specific gravity between the lime and grit, the volumetric proportion of lime to grit worked out in this case to 1 : 2.4, or more lime than that laid down in the bye-laws. With regard to clinker mortars, these were not comprised in the present paper, which dealt only with good brick, grit, or clean, sharp sand, and lime or Portland cement. In the case of mortar made with other materials, other data must be obtained as a basis of calculation.

The presence of soluble silica in cement mortar must not be taken as indicating the actual presence of Portland cement, but that the quantity of Portland cement, if any, could not exceed that indicated by the silica. If there is no soluble silica, no Portland cement could be present.

CHAPTER IX.

MECHANICAL TESTS OF CEMENT.

Specification for Portland Cement, &c.—The following specification for Portland Cement and for Cement Mortar was prepared by Mr. Frederick H. Lewis, and submitted by him for the purpose of discussion at the Engineers' Club of Philadelphia in 1894.*



ON THE MEDWAY.

periods up to at least one year, and also records of analyses, showing proportions conforming to recognised standards, and to the chemical requirements below. Such tests and analyses must be made by competent parties, certified by them to be correct, and, if required, sworn to.

Prior to the award of contracts, parties wishing to be considered will submit samples, duly marked for identification, and each guaranteed to be an average sample of the cement to be furnished in the event of an award. The sample will be used for preliminary tests, and also preserved for comparison with the cement delivered for use.

GENERAL.

(1) *Records and Samples.* — Parties offering cements for engineering works must be prepared to file satisfactory records of tests, both neat and in sand mortars, for

* "Proceedings" Eng. Club, Phila., 1895, xi., p. 325.

(2) *Testing*.—The briquette moulds, sand sieves, setting needles, &c., and the methods of making briquettes and of testing, will all be in accordance with the recommendations of the American Society of Civil Engineers, with this exception, viz., that the cement may be screened through a No. 30 sieve, if necessary, to remove lumps and foreign matter, but other than this, briquettes will be made up from cements as samples without sifting.

Tests for setting lime, fineness, and the seven-day tensile tests required below, will be made from each sample taken. The chemical tests will be furnished by manufacturers before the award of contracts, but will be confirmed by occasional tests of the cement furnished.

The requirements in tensile tests are for an average of a set of five briquettes in each case.

(3) *Fineness*.—Cements will not be considered unless they are ground so finely that $97\frac{1}{2}$ per cent. will pass through the standard No. 50 sieve, and $87\frac{1}{2}$ per cent. through the No. 100 sieve.

(4) *Specific Gravity*.—Cements will be required to have a specific gravity exceeding 3.00.

(5) *Constancy of Volume*.—Mean made neat in pats, $3\frac{1}{2}$ in. in diameter, $\frac{3}{8}$ in. thick at the centre, and drawn to a sharp edge at the circumference, the cements must show no evidence of cracking, checking, or warping when exposed in the air, or in water at normal temperature, for one week.

(6) *Sampling, Storing, and Accounting*.—All tests will be made from average samples selected at random from 10 per cent. of the packages offered. Not more than 150 barrels will be accepted on one set of tests, and no cement will be used which has not been duly sampled and tested at seven days.

Contractors will provide good and sufficient storage for

cements, so that there shall always be an ample stock on hand, and will protect the stock against intrusion, or the use of lots not authorised.

The Inspector will have free access to the storehouse at all times, and will be afforded every facility for identifying and accounting for different lots of cement in store. He will be furnished promptly with duplicate invoices and bills of lading of all cement for the work as shipments are made.

In case of rejection, the lot of cement condemned must be removed from store within twenty-four hours.

(7) *Cost of Testing*.—Parties making propositions for furnishing cements will include in their prices a sum sufficient to cover the cost of having the cement regularly sampled and tested in a manner satisfactory to the purchaser.

(8) *Limits of Accuracy*.—Chemical determinations will be considered accurate to the nearest tenth of a per cent.; tensile tests to the nearest ten pounds. Within these limits tests must meet the requirements of specifications, or the cement will be rejected. There will be no re-tests.

(9) *Cements for Salt Water Exposures*.—Initial set with fresh water, not less than ten minutes.

CHEMICAL.

Sulphuric acid (SO ₃) less than	1'00 per cent.
Magnesia (MgO) less than	1'00 ..

TENSILE TESTS.

1 day neat	not more than 45 per cent.
	of tests at 7 days neat
7 day neat	375 lb.
7 days 3 parts sand to 1 of cement	135 lb

(10) *Quick-setting or Untreated Cements for Fresh water*

Exposures.—Initial set, with fresh water, not less than ten minutes.

CHEMICAL

Sulphuric acid less than	1.25 per cent.
Magnesia, less than	2.50 "

TENSILE TESTS,

1 day neat..	not more than 45 per cent. of tests at 7 days neat
7 days neat	375 lb.
7 days 3 parts sand to 1 cement	150 lb.

(11) *Slow-setting or Treated Cements for Fresh-water Exposures.*—Initial set not less than ten minutes.

CHEMICAL.

Sulphuric acid less than	2.00 per cent.
Magnesia less than	2.50 "

TENSILE TESTS.

1 day neat..	not more than 45 per cent. of tests at 7 days neat
7 days neat	475 lb.
7 days 3 part sand to 1 cement	175 lb.

MORTARS.

(12) *Sand.*—The sand used shall be of such quality that it will give in laboratory tests, 3 to 1 at 7 days, 60 per cent. of the tensile strength required for briquettes made with standard sand, and briquettes made directly from the mortar box will similarly be expected to show 33 per cent. of the test requirements for standard sand.

In laboratory tests of sand the lumps will be removed by sifting through a No. 20 sieve.

(13) *Mixing Mortars.*—The sand must in all cases be

dry, and will be stored in such a way that it can be kept dry. For mortars, sands will be sifted so as to exclude lumps or particles larger than $\frac{1}{8}$ in. diameter; for concrete, to exclude those larger than $\frac{1}{4}$ in. diameter. In proportioning mortars, the cements will invariably be measured by weight; the sand by bulk, to be agreed upon. A thorough mixture of sand and cement will be made dry, after which the water will be added by sprinkling, and the whole mass worked to a temper.

The amount of mortar to be mixed at one time will be fixed to suit the character of the cements. No mortar will be used which has been mixed over night, or over a length of time fixed by the inspector.

(14) *Lime in Cement Mortars.*—In the case of a mixture of lime and cement in mortars, the lime must be slaked at least ten days before using.

(15) *Concrete.*—In making concretes, the cement and sand will first be mixed dry in the manner set forth above, and worked both dry and wet to a temper. The stones measured by bulk will first be sprinkled and then added to the mortar and well worked in.

The concretes will be made with a minimum of water, to admit of being rammed in place without showing an excess of surface water.

Fineness.—In the course of the discussion which followed the reading of Mr. Lewis' paper, Mr. Robert M. Lesley pointed out that the following finenesses are required by the Governments of Europe:—

Belgium	15 per cent. on a 5800 mesh sieve (Genie Belge)
Germany	10 per cent. on a 5800 mesh sieve (Rule Prussian)
Switzerland	15 per cent. on a 5800 mesh sieve (Report Tetmajer, Zurich)

France	No fineness specification (Cahier des Charges, Ponts et Chaussées)
France	5 per cent. on a 5800 mesh sieve (recommended by Le Chatelier and Caudlot, French Chemists)
Rouman'a	12 per cent on a 5800 mesh sieve (Government specification)
England	8 per cent. on a 2500 mesh sieve (recommended by Faija, World's Fair Congress, 1893)
United States	Recommendation of American Society of Civil Engineers, June 27th, 1893, says, "Cements of better grades are ground so fine that only from 5 per cent. to 10 per cent. is rejected on a sieve of 2500 meshes per square inch."

The following results of a series of tests of mortar made with cements of increasing fineness, conducted by Messrs. Colson and Colson * will be of interest :—

Relation of the Fineness of Cement to the Tensile Strength of Mortar in lbs.

Size of Sand.	Fineness of Cement used.			
	As received.	Through 2500 meshes per sq. in.	Through 3600 meshes per sq. in.	Through 5800 meshes per sq. in.
Screened through 2500 meshes per square inch ..	lb. 122	lb. 133	lb. 203	lb. 240

Each result is the average of six experiments. Same weight of cement used in all the experiments.

* "Proceedings" Inst. C.E., Vol. xcv., Part I.

Tensile Strength.—Mr. Lesley quoted the following specifications:—

Neat Cements.

France	7 days, 284 pounds; 28 days, 497 pounds (Cahier Gullian)
Russia	7 days, 255 pounds (Russian Public Works)
Belgium	7 days, 255 pounds; 28 days, 497 pounds. (Genie Belge)
England	7 days, 350 pounds (Faija)
Germany	No neat requirements
American Soc. of C.E.	250 to 550 pounds, 350 to 700 pounds. (tests recommended)

Sand Tests.

3 Sand to 1 Cement.

France	28 days, 215 pounds
Germany	„ „ 227 „
Russia	„ „ 142 „
Belgium	„ „ 215 „
England	None

American Society of C.E. recommends 7 days, 80 to 125 pounds,
28 days, 120 to 200 pounds

Checking.—After three years' discussion, continued Mr. Lesley, the boiling test was finally reported against at the Session 1893, by the German Cement Association, in conjunction with the Prussian Minister (Gray's Paper, World's Fair Congress, August, 1893). Nor is it contained in the most notable French specifications. Faija's Paper, at the World's Fair Congress, August 2nd, 1893, recommended the steaming test only, and immersion at 116 deg. Fah. The American Society of C. E. requires no boiling test.

Mr. Grant's Results.—The following conclusions were arrived at by the late Mr. John Grant, M. Inst. C.E., in the course of his very extensive and exhaustive experiments on the strength of cements:—

(1) The earlier experiments, made six years ago (1859),

gave, at the end of a month, a breaking weight varying from 75 lb. to 719 lb. on an area of $2\frac{1}{4}$ square inches.

(2) A minimum test of, at first, 400 lb., and afterwards of 500 lb., was specified.

(3) During six years the average strength of 1,369,210 bushels used in the Southern Main Drainage Works has been 606.8 lb., being 52 per cent. above the standard first specified, and 21 per cent. above that subsequently adopted.

(4) The average weight per bushel has been 114.15 lb., being 4.15 per cent. above the specified standard.

(5) Portland cement has been proved to be peculiarly suitable for hydraulic works, and may be procured in any quantity and of the highest quality.

(6) Portland cement, if it be preserved from moisture, does not, like Roman cement, lose its strength by being kept in casks or sacks, but rather improves by age—a great advantage in the case of cement which has to be exported.

(7) The longer it is in setting the more its strength increases.

(8) Neat cement is stronger than any admixture of it with sand.

(9) Cement mixed with an equal quantity of sand (as has been the case throughout the Southern Main Drainage Works) may be said to be, at the end of a year, approximately three-fourths of the strength of neat cement.

(10) Mixed with two parts of sand it is half the strength of neat cement.

(11) With three parts of sand the strength is a third of neat cement.

(12) With four parts of sand the strength is a fourth of neat cement.

(13) With five parts of sand the strength is about a sixth of neat cement.

(14) The cleaner and sharper the sand, the greater the strength.

(15) Very strong Portland cement is heavy, of a bluish-grey colour, and sets slowly. Quick-setting cement has generally too large a proportion of clay in its composition, is brownish in colour, and turns out weak, if not useless.

(16) The stiffer the cement mortar, that is, the less the amount of water used in working it up, the better.

(17) It is of the greatest importance that the bricks or stone with which Portland cement is used should be thoroughly soaked with water. If under water in a quiescent state, the cement will be stronger than cut of water. Table XIX. shows that cement kept in water was one-third stronger than that kept out.

(18) Blocks of brickwork or concrete made with Portland cement, if kept under water till required for use, would be much stronger than if kept dry.

(19) Salt water is as safe for mixing with Portland cement as fresh water.

(20) Bricks made with neat Portland cement are as strong at from six months to nine months as the best quality of Staffordshire blue bricks, or similar blocks of Bramley Fall stone, or Yorkshire landings.

(21) Bricks made of 4 parts or 5 parts of sand to 1 part of Portland cement will bear a pressure equal to the best picked stocks.

(22) Portland cement concrete, made in the proportions of 1 of cement to 8 of ballast, in some cases, and of 1 to 6 in others, has been extensively used for the foundations of the river wall, piers of reservoirs, and foundations generally, at Crossness and Deptford, with the most perfect success, and it is believed that it might be much more extensively used as a substitute for brickwork or masonry.

wherever skilled labour, stone, or bricks are scarce, and foundations are wanted at the least expenditure of time or money.

(23) Wherever concrete is used under water, care must be taken that the water is still; as otherwise a current, whether natural or caused by pumping, will carry away the cement and leave only the clean ballast.

(24) Roman cement, though about two-thirds the cost of Portland, is only about one-third its strength, and is therefore double the cost, measured by strength.

(25) Roman cement is very ill-adapted for mixing with sand.

In conclusion, Mr. Grant, whilst recommending Portland cement as the best article of its kind that can be used by the engineer or the architect, warned anyone against its use who is not prepared to take the trouble or incur the trifling expense of testing it, as if manufactured with improper proportions of its constituents—chalk and clay—or improperly burnt, it may do more mischief than the poorest lime.

Mechanical Tests of Portland Cement.—Various machines have been introduced for the purpose of testing the tensile strength of cements by breaking briquettes formed under definite conditions, at stated intervals. The two most commonly employed, viz., that of the late Mr. Henry Faija, constructed by Messrs. Bowes, Scott, and Western, Limited, and that of Messrs. George Salter and Co., of West Bromwich, are described below. The following instructions for testing were drawn up by the late Mr. Faija:—

“*Sampling.*—When it is required to take a sample of cement for testing, it is desirable, in order to secure a fair average sample, to take a small quantity from two or three out of every hundred barrels or sacks, or the equivalent in

bulk, and mix them all well together before taking the quantity required for testing, the samples being taken well from the centre of the sacks, barrels, or bulk, and not from the surface, as that portion may have been accidentally damaged.

“Gauging and Manipulation.”—To obtain the best results, whether in the testing-room or in actual work, the minimum of water should in all cases be used when gauging cement. In the testing-room, small experimental pats should be made with a weighed quantity of cement and a measured quantity of water, in order to determine the exact amount of water required to properly gauge the particular sample under consideration. Having arrived at this knowledge, a sufficient quantity of cement for filling a nest of moulds (five moulds require 30 oz.) should be put into the gauger, and the proper quantity of water added thereto. The handle of the gauger must then be turned until the cement has attained the proper consistency; it should then be turned out of the gauger on to the gauging slate, knocked up with a trowel into a convenient form, and filled into the moulds, being lightly rammed and gently shaken in order to remove all air bubbles. The briquettes should then be smoothed off with a trowel and placed on one side. The whole operation, from the time of putting the water to the cement to placing the briquettes on one side, should not exceed five or six minutes. The briquettes should be removed from the moulds at the expiration of twenty-four hours from gauging, and placed in water, where they should remain until due for testing. It is customary to determine the tensile strength at the different dates by the average of five briquettes at each date.

“Three pats should be made on pieces of glass or other non-porous substance, and their behaviour watched under

the following conditions :—Pat No. 1 may be left in the air, and pat No. 2 should be put in water as soon as it is set. Pat No. 3 should be treated in the apparatus for determining the soundness of cement. The apparatus consists of a covered vessel, in which water is maintained at an even temperature of 110 deg. The space above the water is therefore filled with the vapour rising therefrom, and is at a temperature of about 100 deg. Immediately the pat is gauged it should be placed in the upper part of the vessel, in which racks are provided for the purpose, and in five or six hours it may be placed in the warm water and left therein for nineteen or twenty hours. If, at the end of that period, the pat is still fast to the glass and shows no signs of blowing, the cement may be considered perfectly sound ; should, however, any signs of blowing appear, the cement should be laid out in a thin layer for a day or two, and a second pat made and treated in the same manner, as the blowing tendency may only be due to the extreme newness of the cement. If pat No. 3 shows the cement to be unsound, pats No. 1 and 2 will eventually prove it, but it may be weeks or even months before they develop the characteristics. If pat No. 2 blows, it may be because it was put into the water before it was set.

“ SPECIFICATION.

“ *Fineness.*—To be such that the current will all pass through a sieve having 625 holes (25^2) to the square inch, and have only 10 per cent. residue when sifted through a sieve having 2500 holes (50^2) to the square inch.

“ *Expansion or Contraction.*—That a pat made and submitted to moist heat and warm water at the temperatures and in the apparatus already described, shall show no signs of blowing in twenty-four hours.

“*Tensile Strength.*—Briquettes which have been gauged, treated, and tested in the prescribed manner, to carry an average tensile strain, without fracture, of at least 175 lb. at the expiration of three days from gauging, and those tested at the expiration of seven days from gauging, to show an increase of at least 50 per cent. over the strength of those at three days, but to carry a minimum of 350 lb. per square inch.

“In applying the strain to a briquette when testing for tensile strength, it is important that the strain should be applied evenly and always at the same rate. A difference of 25 per cent. may be obtained by applying the strain very quickly or very slowly.”

In the “Proceedings,” Institution of Civil Engineers, vol. lxxv., page 225, Mr. Faija gives the following:—

“*Summary of Results of Experiments to Determine the Difference Obtained by Applying the Weight to the Briquette, when Testing for Tensile Strength at Different Speeds.*

No. of briquette.	Speed.		Average result.	
	Pounds.	Seconds.	Pounds.	
129	100 in	1	560	75
129	100 in	15	506	43
145	100 in	15	452	20
145	100 in	30	430	96
90	100 in	30	417	27
90	100 in	60	403	05
40	100 in	60	416	75
40	100 in	120	400	87

“From the foregoing results it will be seen that the increase per cent. due to increased speed of applying the strain is as follows:—

“Taking the lowest speed of 100 lb. in 120 seconds as a starting-point, by applying the strain at the rate of

100 lb. in 60 seconds, the increase is 3.960 per cent.

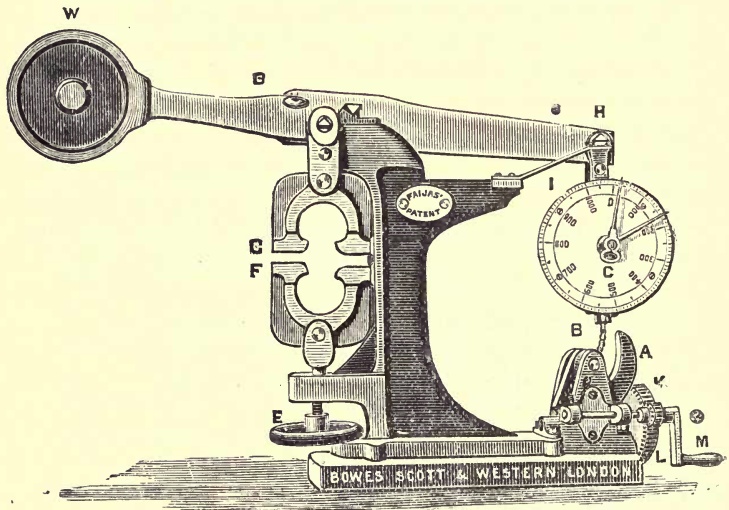
100 lb. in 30 " " " 7.488 "

100 lb. in 15 " " " 12.416 "

100 lb. in 1 " " " 23.142 "

"The standard of speed now adopted is 100 lb. per 15 seconds."

The following is an illustration of Faija's cement-testing machine.



The ordinary-sized machine, adapted to test briquettes of 1 square inch section, will test from 1 lb. up to 1000 lb.; it stands 14in. high, is 14in. long by 3in. wide, and weighs under 30 lb.

The knife edges and wearing parts are of phosphor bronze, and special gearing has been arranged so that the strain may not be put on the briquette at too great a

speed ; 100 lb. per 15 seconds is now considered the standard. The clips of the machine are made to suit the form of briquette adopted by the Metropolitan Board of Works. The clips can be made to suit other forms of briquette as required.

The makers' instructions for using this machine are as follows :—On receiving the machine, clean off all old oil and re-lubricate ; attach the balance weight **W** to the short end of the lever.

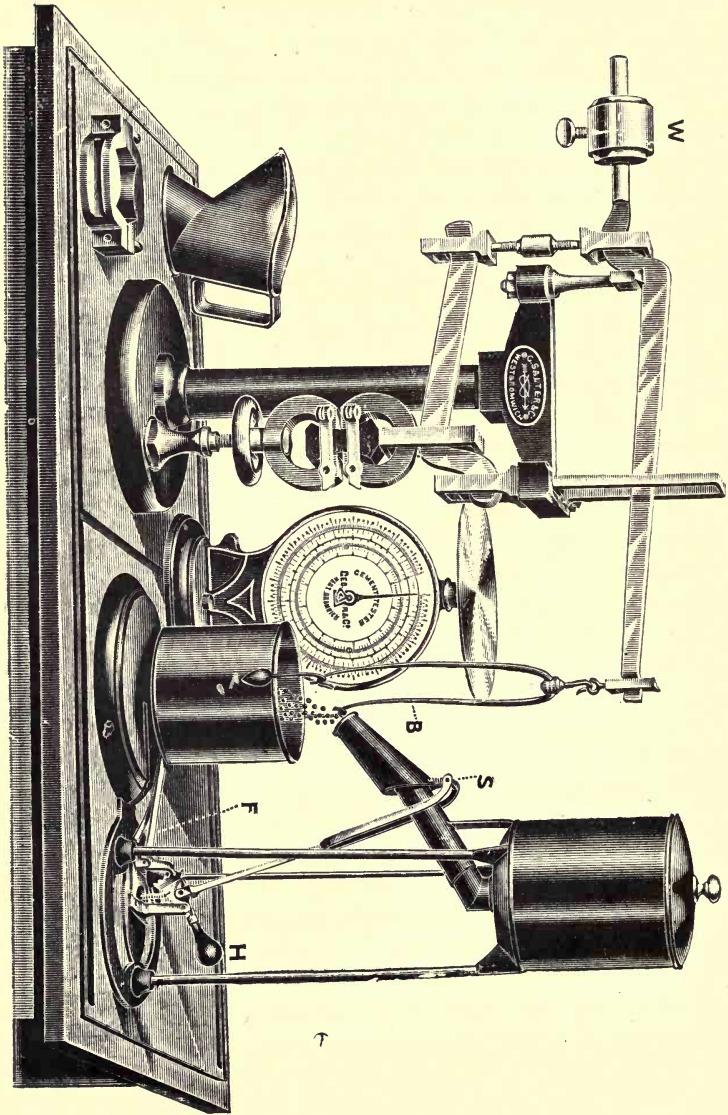
To Use the Machine.—See that the quadrant **A** is in the position shown in sketch, so that the chain **B** to the dial **C** is slack, and the lever **D** free and balanced.

Turn the wheel **E** from right to left, until the lower clip **F** can be raised into contact with the upper clip **G**.

Insert the briquette to be tested in the clips, taking care that it is put in true and evenly, so that the pull on it and the clips is true and vertical ; then turn the wheel **E** from left to right, which will bring down the lower clip **F**, and secure the briquette firmly in the clips. (It is generally advisable to put such a strain on the briquette by turning wheel **E**, that about 100 lb. is indicated on the dial.) When in this position there should be about half an inch between the under side of knife edge **H** and the buffer or recoil spring **I**.

Having seen that the pinion **K** is in gear with the wheel **L**, turn the handle **M** until the briquette breaks. The loose pointer will show on the dial the strain in pounds at which the briquette broke.

To Return to Zero.—Throw the pinion **K** out of gear with the wheel **L** by removing the pin and pushing it to the left ; turn the wheel **L** from left to right until the quadrant **A** has returned to its normal position, with the chain **B** slack ; put the loose pointer back to zero ; release the lower



clip **F** by turning wheel **E** from right to left ; remove the broken briquette, and insert the next that is to be broken.

Messrs. Geo. Salter and Co., of West Bromwich, are the makers of a Compound Lever Cement Testing Machine, of which a woodcut is given on the page opposite.

This apparatus consists of a cast iron column, carrying a pair of compound levers, having a combined leverage of 50 to 1. The levers are fitted with tempered steel knife edges, which rest on polished concave bearings, also of tempered steel, thus obtaining a very sensitive balance. A sliding balance-weight, for the purpose of setting the levers in equilibrium, is fitted to the upper lever. The upper clamp to receive the cement briquette is suspended from a knife edge on the lower lever ; the lower clamp is attached to the base of the column, and is adjustable by means of a screw and a small hand-wheel. The supply of shot to the bucket is automatically cut off at the moment the briquette breaks. To use the machine, set the levers "floating" by means of the sliding balance-weight **W**, then place the briquette in the clamps, hang the bucket on the bridle **B**, and screw down the small hand-wheel until the end of the lever from which the shot bucket is suspended is as high as it can conveniently be ; press down the handle **H** until the shot flows at the desired rate. Immediately the briquette breaks, the bucket into which the shot has been poured falls upon the lever foot **F**, the sliding shutter **S** is released, falls, and cuts off the supply of shot. The bucket and shot are then weighed on a Salter's Spring Balance, fitted with a special dial, on which the breaking strain is indicated without any calculation. Should the shot become blocked, and cease to flow, a slight tap will re-start it. A mixture of shot, No. 6 and No. 10, may be used. The maximum load on the machine should not exceed 1000 lb.

Little is really known yet of the action of percolation of water (fresh, rain, or salt) or paraffin, &c., through various strengths of concrete and under various heads.

To endeavour to find out the most economical concretes for this, Messrs. Hilton and Co. have devised a neat apparatus for the purpose, and easy for anyone to use. It is made solely by Calvert, Harris, and Co., of 54, Cannon Street, London.

It consists of a small hand pump over a cistern, which pumps up an accumulator, the table of which can be weighted to any head desired, and which is read at a glance by the special gauge marked off into feet head and pounds per square inch. This is connected to two gun-metal boxes, heavily ribbed to prevent bulging and leaking when under pressure. The interior of the boxes are also slightly ribbed, to prevent all chance of leakage next the metal. Each box measures a foot cube exactly, and either can be shut off the pressure pipe and be entirely disconnected or not at will. Foot cube blocks can be reported on, or any less thickness, or the top and bottom of the case can be bolted together over any sample paving slab, and results equally well noted. The percolation runs through into the bottom half of box, and is measured in special closed glass containers marked off in cubic centimetres.

The inventors of this machine will shortly have some useful information to impart on percolation, as they are continuing their experiments in this direction.

Two boxes are supplied to enable another sample to be got ready while one is under test, or a different quality sample can be tested at same time in the second box, and so time saved. More boxes can be coupled up to the same accumulator if desired, to still further save time. Under

the best circumstances making and recording these tests is necessarily a slow and tedious process, and requires great care.

CHAPTER X.

THE ADULTERATION OF PORTLAND CEMENT.

THE great importance of cement being thoroughly reliable has naturally led to considerable discussion as to the necessity of this substance being supplied in a pure and sound condition, and the practice which has arisen of adding to good cement various ingredients with a view to its so-called improvement has been sharply called to account.

So injurious to the interests of the manufacturers of good cement has this sophistication been, that in 1894 the cement trade held a meeting at the London Chamber of Commerce* "for the purpose of establishing an association of English cement manufacturers," and "of dealing with, and, if possible, of putting a stop to, the unprincipled and disreputable practice" of "adulterating cement by the mixture of Kentish rag-stone, other stone, furnace or oven ashes, disused or exhausted firebricks, and other inert material." It was attended by representatives of twenty-nine manufacturers of Portland cement. There were considerable differences of opinion on the various points, some approving and others objecting to the proposed association, whilst others preferred to leave the matter in the hands of the London Chamber of Commerce. One gentleman denied that the addition of Kentish rag-stone was an adulteration, and contended that it improved the cement in point of colour and tensile strength, the sand tests were higher, and in every respect it was a better

* *Journal* of the Society of Chemical Industry, December 31st, 1894, page 1236.

article. Another manufacturer said that English cement manufacturers were making a better article than they did some years ago, but admitted that they had been distanced by German skill. He thought they "ought not to be debarred from imitating that skill, from taking advantage of scientific research, and from making all the progress they could; and therefore held that the matter should be sifted by a qualified body in a scientific manner, and then fully reported upon."

"In this connection it is instructive to note," continues the *Journal*, "what the Germans have done in the same direction. According to the *Chamber of Commerce Journal*, early in the year 1880, when adulteration threatened to become very prevalent, the Union of German Cement Manufacturers found itself compelled to set its face in the strongest possible manner against the admixture of foreign materials with cement, in order to protect both their good name as well as the building trade, from disadvantage and danger. On the other hand, the practice was defended as producing a cheaper cement and yet sufficiently strong for that trade. The Union, however, insisted that the question of price must be left to the consumer, who, if he wished, could make the mixtures himself. If manufacturer or dealer were permitted to do this, the door would be open wide to fraud, and the confidence of the public be shattered, for the consumer would not be in a position to know or test either the degree of adulteration or the characteristics of the added materials.

"With regard to the alleged improvement, it was certainly known that by the addition of ultramarine, precipitated silicic acid, preparations of potters' clay, &c., the strength of Portland cement could be increased, but it was still doubtful whether the other valuable qualities of Portland

cement do not suffer, and, moreover, these admixtures are excluded, if only by reason of their high price.

“With the view of rooting out, as far as possible, this system of adulteration, the Union drew up certain information as to what may be legitimately added to cement and what may not. The following were the conclusions arrived at:—

“*Admixture of Gypsum.*—The addition of unburnt gypsum, made at the time of grinding, has for its advantageous object the retarding of the setting of such Portland cements as are by their nature quick-setting, and thereby rendering them more fit for use. The action of the gypsum is probably owing to the fact that when the cement is mixed with water into mortar, the gypsum is first dissolved, and then precipitated in extremely fine particles on the grains of the cement, in exactly the same way as it is recommended for lime by F. Schott (see *Dingler's Journal*, 205, 52, and 209, 30). If the particles of cement are enveloped in the thinnest possible film of gypsum, the chemical incorporation of the water is notably delayed.

“In consequence of this, the cement becomes slower in setting, and by this means (possibly also by the simultaneous occurrence of some chemical action) cement mixed with a little gypsum gives a higher degree of strength than the unmixed quick-setting cement. Inasmuch as the addition of from $\frac{1}{2}$ to 2 per cent. of gypsum is sufficient for the attainment of this object, and as, moreover, the addition of a larger percentage would cause the cement to ‘blow,’ there can be no possible ground for asserting that the admixture of gypsum is made with a fraudulent intention; nay, more, such an addition, within the prescribed limits, must be regarded as an improvement. In consequence of this, the united declaration of the German Portland cement

manufacturers specially permits additions, not exceeding 2 per cent., if made with the object of regulating the time of setting.

“Fraudulent Admixtures.”—Of the materials which may from time to time be mixed with Portland cement with the intention of adding to the profits, and without open acknowledgment of such mixture, it is evident that only those can be employed which are cheap, and which resemble Portland cement both in weight and colour. Chief amongst such materials are slag—principally blast furnace slag—grey limestone, slate, hydraulic lime, black or grey limestone, and trass. The most popular adulterant is slag, because, when pulverised, it is very similar in appearance to Portland cement, and is therefore less easily detected, even when added in large proportions.

“All the foregoing admixtures make the cement inferior, not only in respect of strength, but also in respect of those other valuable qualities (or characteristics) of Portland cement, such as resistance to frost, durability, cohesiveness, &c., as has been proved by the most exhaustive researches.”

As a result of the agitation thus started, the London Chamber of Commerce, on the 30th November, 1894, requested Mr. W. Harry Stanger, M. Inst. C.E., and Mr. Bertram Blount, F.I.C., to undertake the investigation of the effect on Portland cement of the admixture with it of various foreign substances, especially of Kentish rag-stone, and on the 15th May, 1896, those gentlemen presented the final results of their investigations to the Chamber.

As a result of their labours, the Special Committee of the Cement Trade Section of the London Chamber of Commerce carried a resolution to the following effect:—

“That Portland cement be defined as a mixture of two or more suitable materials, intimately and artificially mixed

in the requisite proportions, and afterwards properly calcined and ground, to which nothing has been added during or after calcination, excepting that an addition not exceeding 2 per cent. of gypsum is permissible for the purpose of regulating the setting."

So important, state Messrs. Stanger and Blount, did it appear to the members of the Section to secure Portland cement from adulteration, that they drew up the following declaration, which all manufacturers of Portland cement in Great Britain and Ireland were to be invited to sign:—

"We, the undersigned, hereby agree to conform to and carry out the rule of the Cement Trade Section of the London Chamber of Commerce, as set forth in a report made by the Section, and adopted at a meeting held on Monday, the 10th May, 1897:—

"That if any material whatever, excepting an amount not exceeding 2 per cent. of gypsum for the purpose of regulating the setting, be added to Portland cement clinker, during or after calcination, the article so produced shall not be sold as Portland cement, but under some other distinctive name.

"And we further agree that if at any time any of the parties to this agreement shall, by resolution of a majority of those present at a meeting of such parties duly and properly convened in accordance with the practice of the London Chamber of Commerce, such resolution having been duly and properly confirmed by a majority of those present at a subsequent meeting, called at not less than fourteen days' notice, be found to have failed to conform to and carry out the said rule, then in such case his or their name or names shall be struck off the list, and notice of the same made public in such manner as shall be resolved."

This placed the matter beyond controversy, but in order

that my readers may understand the reasons for this decisive step, I am permitted to append the following substance of Messrs. Stanger and Blount's report to the Chamber of Commerce, a *précis* of which was published in the form of a paper read before the Society of Chemical Industry on November 1st, 1897, above referred to, as follows :—

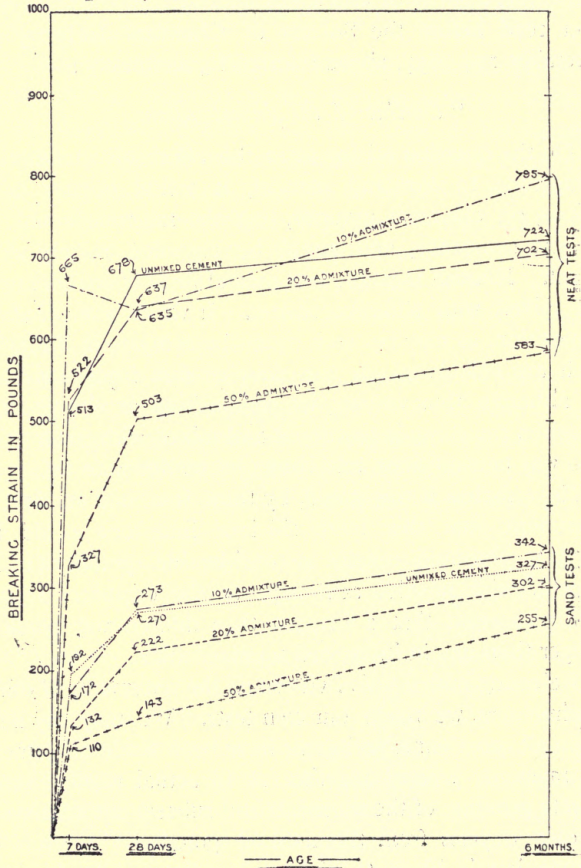
“ Outline of the Investigation.

“ On the question arising as to the effect of an addition of ragstone to Portland cement, it would appear fairly obvious, on chemical grounds, that the ragstone would be an inert substance, and could be regarded merely as a diluent. Kentish ragstone is a natural form of carbonate of lime mixed with siliceous matter. It varies somewhat in composition, as may be seen from the analyses in Table I., which are calculated on the samples free from moisture. But although thus varying, its composition is always such that when the stone is powdered and mixed with water it is in no way cementitious, and shows no tendency to set.

‘ Nevertheless it was stated, by those that had made the experiment, that this inert, silicious limestone could be added to Portland cement in considerable quantity (10-20 per cent.) with positive advantage to the cement. It was credibly asserted that a cement thus diluted was actually stronger than one in its normal unmixed state.

“ Rejecting both *à priori* views and the statements of other experimenters, we made our own tests. We may say here that in all cases our experiments have been made on cement and ragstone, ground together as in actual manufacture; separate grinding of the materials and subsequent admixture not only departs from the method practised by users of ragstone, but is likely to yield misleading results, because the intimacy of admixture is insufficiently great. Mixtures

DIAGRAM OF TENSILE TESTS. SERIES "A."



containing 10, 20, and 50 per cent. of ragstone were prepared in this manner, and tested at seven and twenty-eight days, and at six months, neat and with sand, in tension and compression, and compared with corresponding tests made on the unmixed cement. The diagram on page 108 is typical of certain of the results obtained.

“The composition of these samples is stated in Table II.

“Many similar tests proved beyond doubt that some cements not only do not show a decrease in strength when mixed with 10 or 20 per cent. of ragstone, but are actually stronger when thus mixed. This is true whether the cement and mixtures are tested neat or with sand, in tension or in compression. Thus the upshot is in direct contradiction to what would certainly be expected by anyone endeavouring to solve the question on first principles. The result constitutes a useful example of the danger of making even what appear to be most reasonable assumptions, when it is open to one to dispense with assumptions altogether, and to substitute sound experimental data.

“After close study, the reasons for this apparent anomaly were discerned. It became clear that when ragstone containing, as it commonly does, a small quantity of moisture, is mixed with cement clinker and passed, together with that, through the stone-breaker and millstones, or other grinding machinery, it is brought into the closest possible contact with the cement, and slakes any overlimed portions which may be present. Thus, a cement containing ragstone as it comes from the mills is more slaked than one free from ragstone. Now it is known that cement which is not perfectly sound is improved by limited slaking; it is on account of this that air-slaking or aëration is insisted on by many large users (notably by the Admiralty) in order to hydrate any uncombined lime or unstable lime compounds,

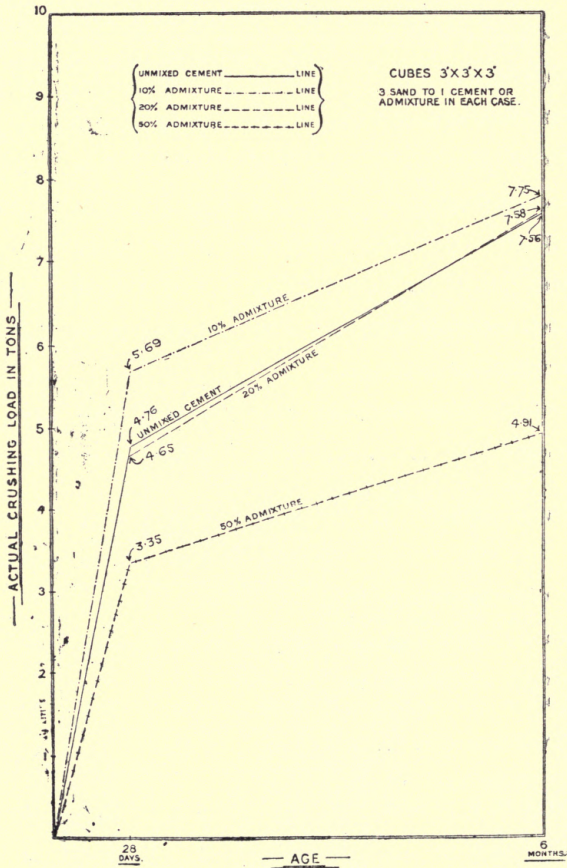
TABLE I

	"A."	"D."	"F."	"L."	"J."
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Silica (SiO ₂)	0.45	0.60	0.08	0.60	0.61
Insoluble residue (sand and clay)	13.53	15.06	22.16	15.60	15.61
Alumina (Al ₂ O ₃)	0.44	0.80	0.14	0.54	0.44
Ferric oxide (Fe ₂ O ₃)	1.22	1.20	1.40	1.30	1.12
Lime (CaO)*	46.84	45.60	41.80	45.42	44.80
Magnesia (MgO)	0.36	0.50	0.44	0.61	0.38
Sulphuric anhydride (SO ₃)†	Nil	0.30	0.16	0.18	0.30
Matter volatile at a red heat:—					
Carbonic anhydride (CO ₂)	35.96	34.30	33.10	33.64	35.30
Water (OH ₂)	0.65	1.14	0.46	1.98	0.92
Alkalies: Potash (K ₂ O), Soda (Na ₂ O), and loss	0.55	0.50	—	0.13	0.52
Total	100.00	100.00	100.00	100.00	100.00
*Value after deduction of that necessary for the formation of calcium sulphate	—	45.39	41.69	45.29	44.59
Corresponding with calcium carbonate (CaCO ₃)	81.73	77.95	75.23	76.45	80.23
†Calculated as calcium sulphate	—	0.51	0.27	0.31	0.51
Specific gravity	2.720	2.680	2.680	2.690	2.685

TABLE II.¶

	Un- mixed cement.	10 per cent. admix- ture.	20 per cent. admix- ture.	50 per cent. admix- ture.
ica (SiO ₂)	Per cent. 20·32	Per cent. 18·36	Per cent. 15·54	Per cent. 10·98
Insoluble residue (sand & clay)	0·70	2·34	4·28	7·54
Alumina (Al ₂ O ₃)	8·36	7·16	6·22	4·72
Ferric oxide (Fe ₂ O ₃)	4·34	4·18	3·80	2·80
Lime (CaO)	59·46*	58·72*	56·62*	53·10*
Magnesia (MgO)	1·04	1·02	1·02	0·83
ulphuric anhydride (SO ₃) ..	1·37†	1·26†	1·11†	0·75†
Matter volatile at a red heat:				
Carbonic anhydride (CO ₂)	1·77	4·90	9·09	17·42
Water (OH ₂)	1·81	1·64	1·81	1·34
kalis: Potash (K ₂ O), soda (Na ₂ O), and loss	0·83	0·42	0·51	0·52
	100·00	100·00	100·00	100·00
*Value after deduction of that necessary for the forma- tion of calcium sulphate..	58·5	57·84	55·84	52·58
Calculated as calcium sul- phate	2·33	2·14	1·89	1·27
Specific gravity	3·075	3·060	2·975	2·920
FINENESS.				
esidue on 76 × 76 sieve ..	8½	9	8½	8
,, 50 × 50 ,, ..	4½	4	4	4
,, 25 × 25 ,, ..	½	½	½	¼

DIAGRAM OF COMPRESSIVE TESTS. SERIES "A."



and prevent their subsequent hydration and destructive expansion when the cement as a whole is gauged with water for preparing concrete or mortar. A moist ragstone, ground

NEAT TESTS.

Breaking Weight in Pounds of Briquettes having 1in. x 1in. Section.

Unmixed Cement.		10 per cent. Admixture.	
7 days.	21 days.	7 days.	21 days.
520	550	480	510
490	540	470	500
460	530	410	480
Average 490 lb.	Average 540 lb.	Average 453 $\frac{1}{3}$ lb.	Average 496 $\frac{2}{3}$ lb.
20 per cent. Admixture.		50 per cent. Admixture.	
7 days.	21 days.	7 days.	21 days.
410	410	240	300
390	410	220	290
380	390	220	250
Average 393 $\frac{1}{3}$ lb.	Average 403 $\frac{1}{3}$ lb.	Average 226 $\frac{2}{3}$ lb.	Average 280 lb.

with such a cement, performs this function of limited hydration, and thus subjects the cement to what is, in effect, an artificial and rapid aëration. Cements which are not perfectly sound are improved by this procedure. But when a

cement is rendered perfectly sound by aëration, this improvement disappears, as is evident from the diagrams given on pages 118 and 119.

SAND TESTS

Breaking Weight in Pounds of Briquettes having 1 in. × 1 in. Section.

Unmixed Cement.		10 per cent, Admixture.	
7 days.	21 days.	7 days.	21 days.
300	330	250	330
260	320	240	290
250	280	—	—
Average 270 lb.	Average 310 lb.	Average 245 lb.	Average 310 lb.

20 per cent. Admixture.		50 per cent. Admixture.	
7 days.	21 days.	7 days.	21 days.
240	300	130	200
230	270	100	160
—	—	—	—
Average 235 lb.	Average 285 lb.	Average 115 lb.	Average 180 lb.

“These figures show that in an aërated, and therefore perfectly sound cement, the ragstone acts precisely as would be expected, viz., as a mere diluent. The effect is the more striking because these very samples before aëration showed

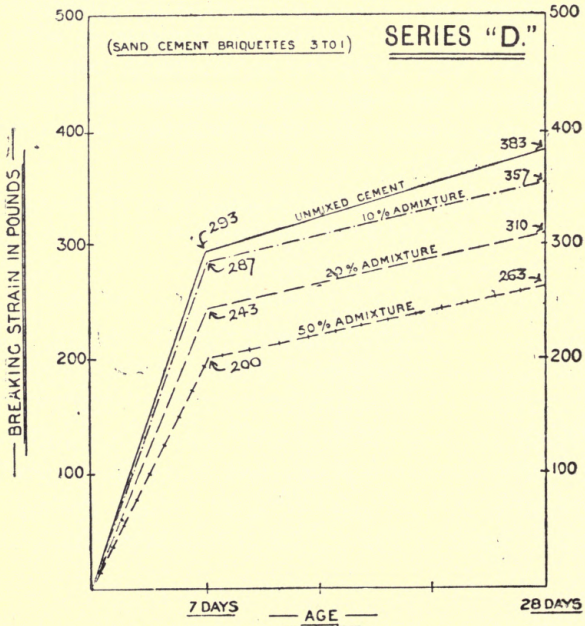
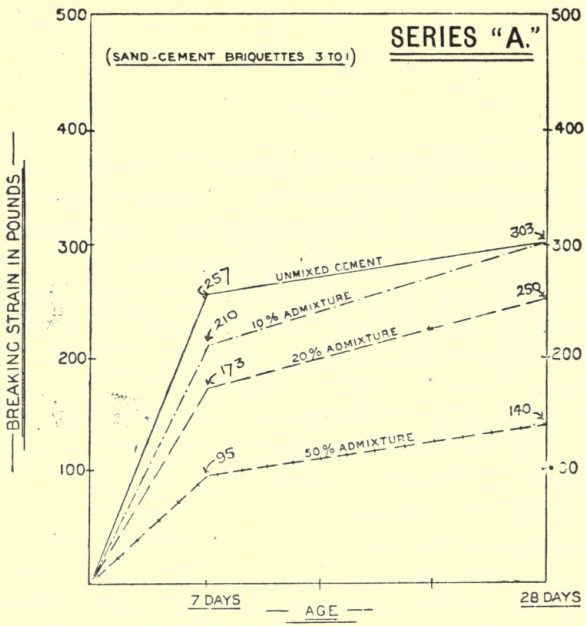
a smaller strength unmixed than when mixed with ragstone. That this alteration of properties is caused merely by the increased soundness of the cement, which is induced by aëration, is proved by the tables on pages 115 and 116.

“In this case the cement used was not aërated by long storage, and yet gave tests which were considerably lower for the mixtures containing ragstone than for the unmixed cement. It was not a cement made in the laboratory, and therefore of better grade than can be readily manufactured on a large scale, but was simply an ordinary commercial cement of high quality and perfect soundness.

“A word must be said as to what is meant by the word ‘soundness,’ which has been used in the previous paragraphs. A cement may appear perfectly sound to ordinary tests, and yet after setting may develop internal stresses which, though not sufficient to cause it to disintegrate or show any sign of actual failure, may diminish its strength appreciably. That this is not an uncommon condition is clear from the fact that many cements are considerably stronger after aëration than when fresh, as is evident from the following table:—

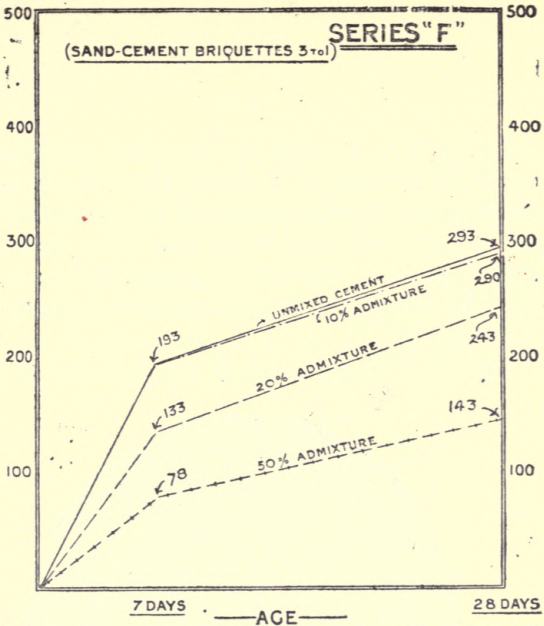
*Breaking Weight in Pounds of Briquettes, 1in. × 1in. Section.
Unmixed Cement and Sand (3 to 1).*

	7 Days.		28 Days.	
	Fresh.	Aërated.	Fresh.	Aërated.
Series "A"	191 $\frac{2}{3}$	256 $\frac{2}{3}$	270	303 $\frac{1}{3}$
.. "D"	259 $\frac{1}{3}$	293 $\frac{1}{3}$	276 $\frac{2}{3}$	383
.. "L"	131 $\frac{2}{3}$	243 $\frac{1}{3}$	190	340

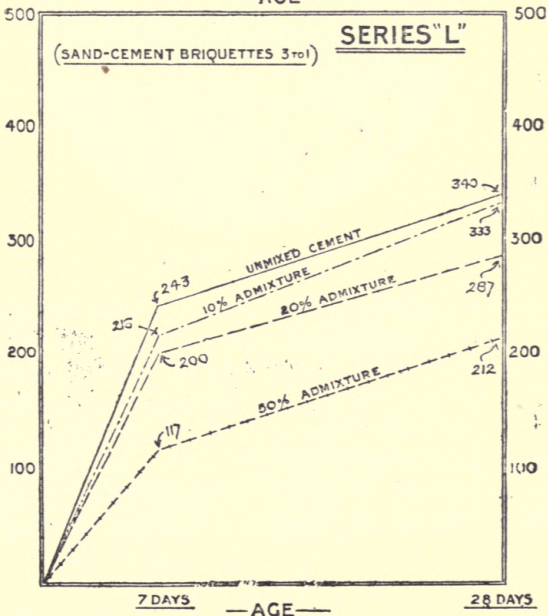


TENSILE TESTS AFTER AERATION.

BREAKING STRAIN IN POUNDS.



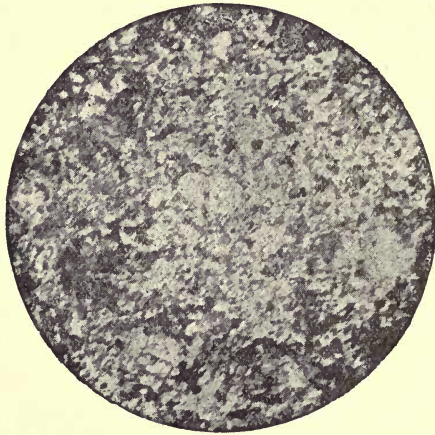
BREAKING STRAIN IN POUNDS.



ENSILE TESTS AFTER AERATION.

“Incidentally, it may be remarked that this great increase in strength is a strong argument in favour of systematic and thorough aëration of all cement that is to be used in heavy and important work. This has long been practised by many engineers to ensure safety; but if the cement be even slightly unsound, a large increase of strength will also accrue.

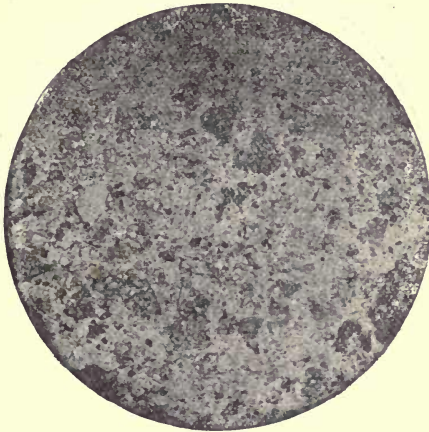
“Reverting to the question of the difference of strength



UNMIXED CEMENT.

of unmixed cement and cement mixed with ragstone, it will be observed that although experiment has established that sound cement is not improved by the addition of ragstone, but is, on the contrary, deteriorated, yet the decrease of strength is smaller than that which corresponds with the percentage of diluent added. Thus a mixture of cement with 10 per cent. of ragstone is not necessarily 10 per cent. weaker, although the ragstone is merely a chemically inert.

addition The reason for this discrepancy becomes clear on studying the structure of set cement. For the purpose of this study, briquettes made from unmixed cement and from cement mixed with 10, 20, and 50 per cent. of rag-stone were chosen, and very thin sections were prepared from them in the manner usual in petrological research. These were examined under the microscope, and a large difference in structure was at once apparent.*



10 PER CENT. ADMIXTURE.

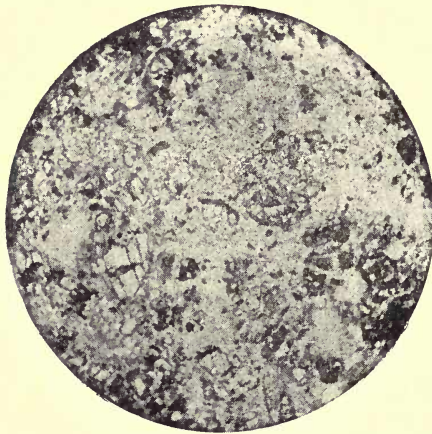
“The microscopic section of the unmixed cement consists of white, nearly opaque, particles, interspersed with dark patches fairly evenly distributed. Most of these dark patches are actual gaps, but some are merely plates of

* The preparation of these photo-micrographs was kindly undertaken by Mr. Herbert Jackson, of King's College, London, to whose skill the beauty and accuracy of the original photographs are sufficient witness.

transparent crystalline substances, which, as they absorb little or no light, appear as gaps in the photograph.

“A similar section of a briquette containing 10 per cent. of ragstone has a much closer structure. Very few true gaps appear in it, the interstices visible in the unmixed cement being filled with fine particles of ragstone.

“With 20 per cent. of ragstone a similar filling is per



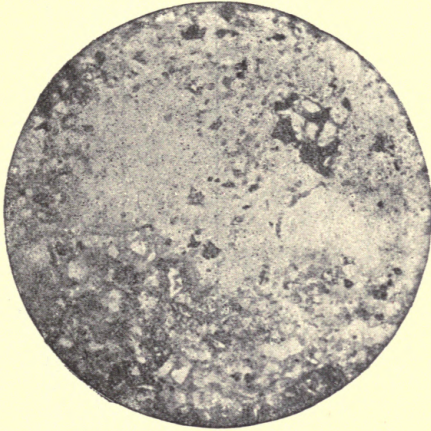
20 PER CENT. ADMIXTURE.

ceptible, but there is also visible a greater proportion of large crystals, either transparent or opaque.

“With 50 per cent. of ragstone the filling of interstices is still noticeable, but the cement is, as it were, swamped with ragstone, and the section contains large inert crystalline masses, which often exhibit cracks, and may be considered as reducing the strength of the material. The appended photo-micrographs illustrate these differences of structures.

“That the greater closeness of structure apparent in the

microscopic sections of the mixtures has a physical existence, was proved by making actual measurements of the total cubic content of the cavities in the set cement and in



50 PER CENT. ADMIXTURE

the mixtures. A maximum density occurs with the 10 per cent. mixture, as is clear from the appended figures:—

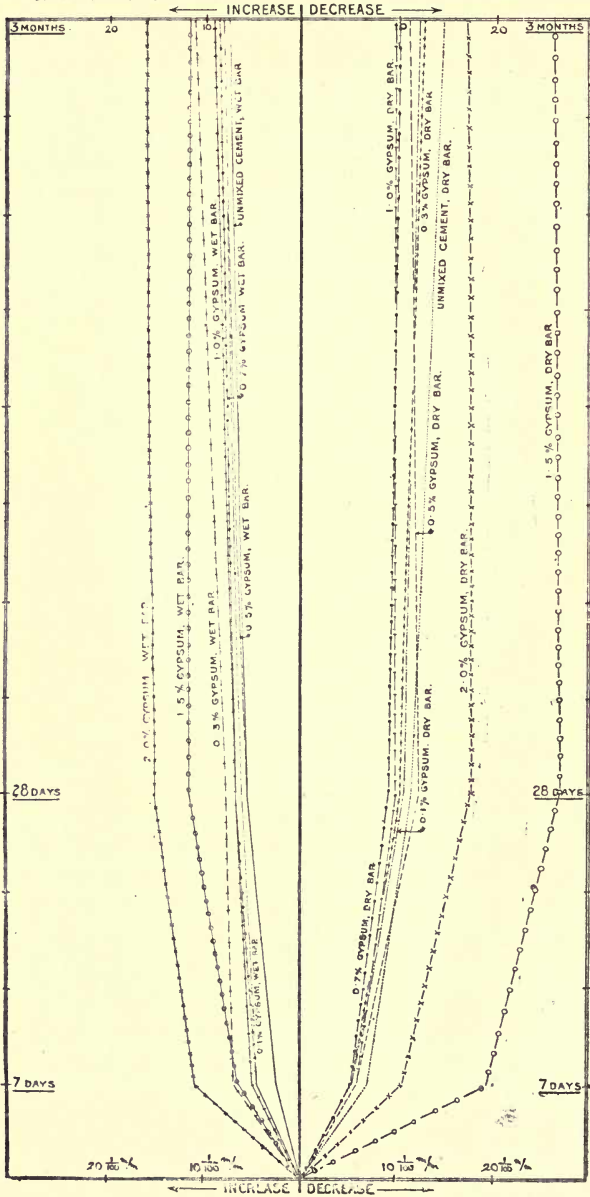
Volume of Cavities in a Briquette, stated in per cent. of the total apparent Volume of the Briquette.

	Series 1.	Series 2.
	Per cent.	Per cent.
Unmixed cement	4·63	2·39
10 per cent. mixture	3·83	1·80
20 „ „	6·32	3·79
50 „ „	10·43	6·32

TESTS FOR CONSTANCY OF VOLUME. SERIES "I."

WET BAR SHOWN BY LINES.
ON LEFT HALF OF DIAGRAM

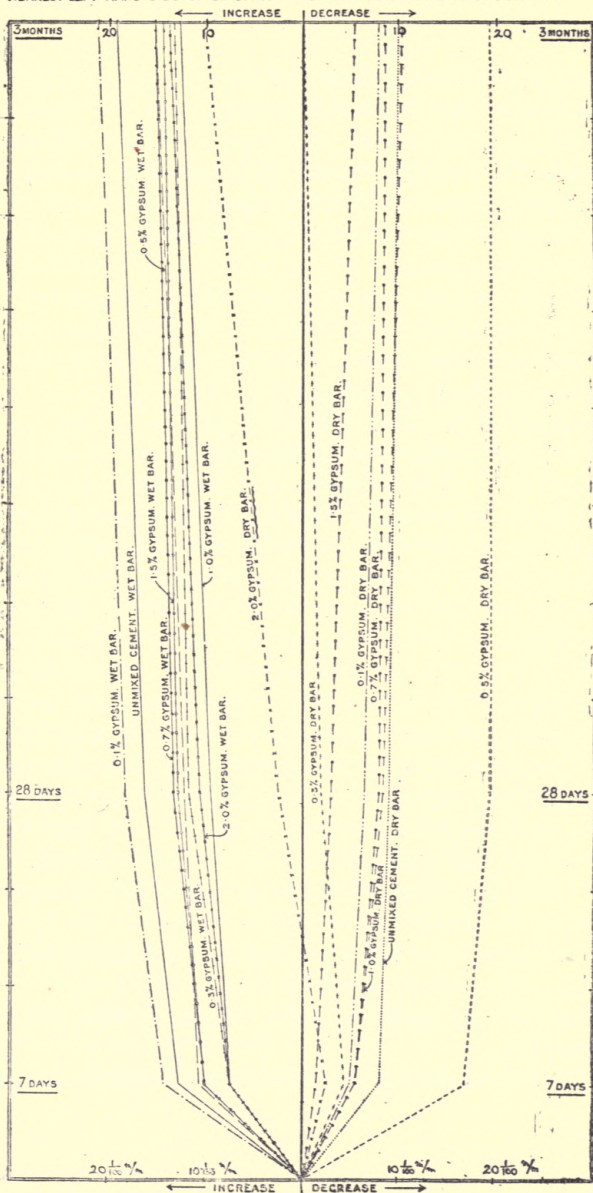
DRY BAR SHOWN BY LINES.
ON RIGHT HALF OF DIAGRAM.



TESTS FOR CONSTANCY OF VOLUME. SERIES "II."

WET BAR SHOWN BY THE 8 LINES
NEAREST LEFT HAND SIDE OF DIAGRAM

DRY BAR SHOWN BY THE 8 LINES
NEAREST RIGHT HAND SIDE OF DIAGRAM



It will be seen that although the large quantity of ragstone in the 50 per cent. mixture communicates to it a loose and porous structure, yet the 10 per cent. mixture perceptibly exceeds the unmixed cement in closeness of texture, this fact serving to confirm the microscopic observations.

“From both microscopical examination of the structure, and from measurement of the cavities in briquettes of unmixed cement and of cement mixed with 10, 20, and 50 per cent. of ragstone, it is evident that a mixture containing 10 per cent. of ragstone has a closer structure than any of the others, even than unmixed cement. The ragstone, in fact, during grinding, yields a quantity of fine powder which serves as a ‘filling’ between the crystals formed when the cement sets. The absence of interstices thus caused appears to compensate in some measure for the fact that the ragstone is inert, and thus a cement containing 10 per cent. of what is simply a diluent is not necessarily 10 per cent. lower in strength than an unmixed cement. In fact, a briquette, even of neat cement, may be regarded as a concrete in which the strength depends not only on the true adhesive and cohesive quality of the cement, but also on the exact fit of the particles of cement and those of the inert matter, such as the coarse core. Any fine inert filling material may improve the closeness of texture of such a concrete if the average size of its particles be such as to occupy fully the spaces naturally present in set cement.

“The whole question concerning the addition of ragstone may be summed up very briefly :—

“1. Ragstone is not a cementitious substance, and its addition to cement is an adulteration.

“2. Perfectly sound cement is weakened by the addition of ragstone.

“3. This weakening is not fully proportional to the per-

centage of ragstone added, because the latter acts as a fine-filling material and fills up the interstices naturally present in set cement.

“4. Cement which is not perfectly sound, may be temporarily improved by the addition of ragstone. When the cement has become sound by aëration, this improvement disappears.

“Many minor points were examined and determined in the course of the main investigation, but the most important results are embodied in the conclusions given above.

“*Additions to Cement other than Ragstone.*

“One of these, which particularly came within our purview in the course of our investigation for the London Chamber of Commerce, is gypsum. Gypsum is used largely in Germany, and to a considerable extent in this country, in quantities not exceeding 2 per cent., and usually smaller than this, in order to lengthen the setting time of the cement. Regarding cement as a chemically finished product in the state in which it comes from the kilns, needing nothing but mechanical comminution to make it saleable, the addition of any substance to the finished clinker must be considered, in strictness, an adulteration. Thus gypsum becomes under this definition an adulterant. Nevertheless it is added for a distinct and useful purpose, and in quantities smaller than 2 per cent. does not affect the cement injuriously, as far as our own experiments indicate. The average results obtained with cement containing proportions of gypsum increasing from 0·1 to 2·0 per cent. are given in the table on page 128.

“It will be seen that no harmful effect on the strength of the cement can be detected.

“The effect of the gypsum on the constancy of volume of

the cement was also determined by the measurement of test bars of unmixed cement and of cement mixed with 0·1 to 2·0 per cent. of gypsum in a Bauschinger apparatus. The results are recorded in the appended curves. On the whole, a slightly greater tendency to expansion is perceptible when the proportion of gypsum exceeds 1 per cent., but the difference is not so great as to warrant the conclusion that gypsum causes any dangerous amount of expansion

Breaking Weight of Briquettes, 1in. × 1in. section (28 Days).

	Sample I.	Sample II.
	Lb.	Lb.
Unmixed cement	618	800
0·1 per cent. gypsum	587	693
0·3 " " " " " "	630	750
0·5 " " " " " "	623	777
0·7 " " " " " "	590	813
1·0 " " " " " "	690	823
1·5 " " " " " "	657	890
2·0 " " " " " "	613	860

“With these facts before us, it is evident that though gypsum is an alien addition to Portland cement, and therefore technically an adulterant, its use can be defended on the ground that it confers specific properties on the cement, and does not affect its strength or soundness unfavourably. The difficulty arising in the question whether cement containing a small quantity of gypsum, added for the purpose of regulating the setting time, can be legitimately sold as Portland cement, has been met by the Cement Trade

Section of the London Chamber of Commerce, by the adoption of the rule which has been quoted above. This rule (page 108) expressly excepts gypsum in quantities up to 2 per cent. from the category of substances which, if added to Portland cement, constitute an adulteration. It will be noted that this convention is similar to that which has been in use for some years in Germany, where it appears to have worked satisfactorily.

“The last and worst adulterant with which it is our purpose to deal is blast-furnace slag. As far as our experience goes, this most objectionable addition to Portland cement is not employed on the Thames and Medway, but in certain other districts it is used in large quantities for the preparation of a grossly sophisticated product which is fraudulently sold as Portland cement.

“We must not be understood as condemning true slag cement made by mixing granulated blast-furnace slag with slaked lime and sold under its proper title. This material is a perfectly legitimate product, and has its own uses; no one can reasonably object to its utilisation if it is not covertly substituted for Portland cement. But the addition of blast-furnace slag to Portland cement is another matter altogether. The general practice of the manufacturers who seek to increase their profits by the use of slag, appears to be to add to the clinker as it goes to the crushers as much crude blast-furnace slag as they consider can be mixed with Portland cement without risk of detection by the ordinary consumer, who buys cement in quantities so small that the cost of its analysis is too great for him to pay. The quantity added may be as much as 30 or 40 per cent.; and detection is not easy, or even always possible, for an unskilled observer. Apart from the fraudulent character of this addition, about which no doubt can well be entertained,

there arises the question of its effect on the cement. And here it is necessary to make a small digression into the chemistry of the subject.

“When Portland cement sets, a certain quantity of lime in the hydrated state is liberated. This lime in cement mortar, or concrete of fair closeness of structure remains distributed throughout the mass, and is there slowly converted into calcium carbonate. Now it is very possible that this lime could be utilised more effectively if it were provided with a certain quantity of silica, or an active silicate with which it could unite in manner similar to that of the lime of a puzzuolanic cement. Further, granulated blast-furnace slag will act as a puzzuolana and unite with slaked lime when the dry mixture of the two substances is gauged with water. It is therefore conceivable that granulated blast-furnace slag could be added to Portland cement in such quantity that its active silicates would unite with the lime set free in the normal setting of the cement. Whether this union would be advantageous to the strength of the cement is a matter for experiment. But whether it is or not, a cement thus dosed with granulated blast-furnace slag could not be legitimately termed Portland cement, and would have to be sold under a distinctive name. It may be mentioned incidentally that ordinary slag contains a good deal of sulphur (*e.g.*, 1 per cent.) in the form of calcium sulphide. This, slowly oxidising, would be likely to expand in the mass of the set cement and cause stresses, which could hardly fail to be injurious, and might be positively dangerous. Thus the burden of proof that the addition of granulated slag to cement is not actively harmful, rests upon the advocates for its use, and even if they prove their case they are confronted by the fact that the mixture is not and cannot be Portland cement.

“ But when the slag added to Portland cement is not granulated blast-furnace slag of the best composition for acting as a puzzuolana, but is the common stony material run out into trucks and allowed to cool spontaneously, instead of being rapidly chilled, the objection to the addition of slag is even stronger. In the first place, this stony, slowly-cooled slag is usually not of such a composition as to allow it to act as a puzzuolana. Next, even if it were, its condition—*i.e.*, annealed by slowly cooling instead of chilled by quenching—is most unfavourable to its puzzuolanic activity. Thus its tendency to unite with lime liberated by the setting of Portland cement would be likely to be small. It may be regarded for practical purposes as a diluent and makeweight. But this is not all; for much of this slag contains a notable proportion of sulphur, and the objectionable effect mentioned above, of the slow oxidation of this sulphur, holds equally in this case.

“ It is evident that the addition to Portland cement of blast-furnace slag, as usually practised, is not only an adulteration, but it is also an adulteration with a dangerous ingredient. Fortunately, this form of fraud is readily detected by analysis, although often escaping recognition by the usual mechanical tests.

“ In conclusion, we may reiterate our views as clearly and briefly as possible.

“ All materials added to Portland cement after the clinker comes from the kilns are adulterants, with the exception of gypsum, which is a recognised addition for a specific purpose in quantities not exceeding 2 per cent. Of the two adulterants which have been specially dealt with, *viz.*, ragstone and blast-furnace slag, the latter is by far the more objectionable, and it should be condemned and rejected by makers and users alike. In this view we believe we are

Tensile Strength in kilos per square inch.

		Pure cement.	2 6 Cement. Normal sand.	1 1 6 Cement. Trass. Normal sand.	1 1 6 Cement. American volcanic ash. Normal sand.	1 1 6 Cement. Swedish factor waste. [†] Normal sand.	
		Water, 25 per cent.	Water, 8½ per cent.	Water, 10 per cent.	Water, 10½ per cent.	Water, 11½ per cent.	
Erdmenger.	7 days	283	123	75	79	160	
	1 month	359	153	119	143	453	
	3 months	447	207	163	202	294	
	6 months	—	235	197	237	330	
	12 months	—	265	222	453	331	
		Water, 23½ per cent.	Water, 12 per cent.	Water, 12½ per cent.	Water, 12½ per cent.	Water, 13½ per cent.	
Lundteigen.	7 days ..	{ F ..	415	125	58	47	260
		{ S ..	485	124	67	74	255
	1 month	{ F ..	496	177	150	139	357
		{ S ..	611	206	185	217	414
	3 months	{ F ..	635	259	194	218	428
		{ S ..	720	286	339	363	469
	6 months	{ F ..	638	304	232	347	459
		{ S ..	824	310	407	419	490
	12 months	{ F ..	638	347	295	399	536
		{ S ..	783	366	421	428	524

N.B.—F = fresh water; S = salt water, containing CaSO_4 1.0214; MgSO_4 , 0.238; NaCl , 0.1515 grm. per litre.

[†] SiO_2 , 59.04; $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$, 14.66; CaO , 1.33; SO_3 , 2.66; loss on calcination, 21.76 per cent.

supported by the great majority of engineers and manufacturers."

Erdmenger and Lundteigen* have published the tables on page 130 showing the tensile strength in parallel experiments of cements with various admixtures with both fresh and salt water.

* *Thouind. Zeit.*, 22 [81], 929-930. *Journal, Soc. C.I.*, vol. xvii., p. 1049.

CHAPTER XI.

THE STRENGTH OF BRICKWORK.

THE most recent information as to the strength of brickwork is that contained in a series of three most valuable reports presented by the Science Standing Committee, to the Institute of British Architects during the years 1896—7. The student who desires to study these in detail will find much valuable data for digestion therein, as well as in the discussion of the reports by the members of the Institute. For the present purpose it will suffice to give the salient features and general outcome of the long series of tests which were conducted by a most able Sub-Committee, consisting of Messrs. Burrows, Street, Unwin, Max Clarke, Garbutt, and Bernard Dicksee; the principal part in the experiments being undertaken by Messrs. Street, Clarke, Garbutt, and Bernard Dicksee. The experiments were confined to the following varieties of bricks, as affording types of the several kinds most generally employed, viz. :—(1) London stocks. (2) Gault. (3) Leicester red. (4) Staffordshire blue. In the third series of tests Fletton bricks. Two piers of each kind, 6ft. high and 18in. square, were built, both with lime and cement mortar, for crushing at the end of three months, and a similar number for crushing at the end of nine months.

The reason for deciding upon having tests at two different periods was to ascertain what additional strength the brickwork gained in six months, as this is of importance, considering the great rapidity with which brick buildings are now run up, and sometimes loaded with great weights while the brickwork is quite green, and very little of the mortar set.

The experiments were conducted upon a vacant piece of ground in front of the engineer's office at the West India Docks, and close to the hydraulic engine-house, from which water at a pressure of 700 lb. to the square inch was available; a very powerful hydraulic press, together with a large and massive testing machine, placed at the disposal of the Committee by Sir William Arrol.

The work of building the experimental piers commenced on the 24th July, 1895, and was finished on the 10th August. They were built upon a temporary line of rails of about 140ft. in length. In the centre of this line of rails Sir William Arrol's testing machine was erected. The piers were built upon wrought iron plates 2ft. by 2ft. 6in. by $\frac{1}{2}$ in., placed upon the rails 1ft. apart, and having two holes drilled in the end nearest the testing machine, by means of which they were drawn along the rails to the machine.

The bricks, lime, and sand were such as would be supplied to any builder in the ordinary way, while the Portland cement was of ordinary quality. The cement mortar was mixed by measure in the proportion of 1 of cement to 4 of washed river sand, and the lime mortar 1 to 2. The brickwork started upon a bed of mortar of the thickness of an ordinary joint, and rose, as nearly as possible, four courses to the foot. The bond was the same as that shown in Rivington's text-book, and was the ordinary way of building an 18in. pier.

As showing the great difficulty of controlling the operations of the men employed in the work, the Committee stated with regret, that during the absence of members of the Committee, the bricklayers, finding the Leicester red and the Staffordshire blue bricks very hard to cut, filled in a great portion of two piers with closers of London stocks.

This quite destroyed the value of these piers, and put the Committee to the expense of building fresh piers to take their place. This unintentional outcome of part of the experiments is, however, not without its value, as indicating the care which should be exercised in controlling the work of the men employed.

The following extracts from notes by Mr. Matt. Garbutt on the behaviour while under compression, of the first series of brickwork piers, will indicate the character of the results obtained.

The observed effects of pressure were similar in most cases, but varied in degree with the different materials. Generally, the first evidences of a strain were slight internal crackling sounds, audible only on applying the ear to the brickwork. Then the mortar squeezed out of the joints and fell off—this occurring to a greater extent with the harder bricks, and being more marked where lime mortar was used than with cement. This was accompanied or followed by cracks occurring in single bricks, which, from slightly irregular bedding or other causes, were unevenly stressed, and by the spalling off of small corners or pieces of the face for similar reasons. By this time the joints were, generally, noticeably compressed, and serious cracks, indicating final lines of rupture, appeared. The piers at this point generally bulged outwards—slowly at first, and then, the resistance of the brickwork ceasing, the falling of the pressure gauge gave warning that actual collapse was imminent. In the case of the harder bricks, set in cement, the final failure was much more sudden than in the others, so much so that some agility was necessary on the part of the man who, to measure its compression, stood close to the pier.

From the beginning of the tests, all through the series, it was evident that the vertical line of joints formed by the

closers was a plane of weakness, and it was generally at this line that the serious cracks first showed themselves. The majority of the piers broke by crushing in the normal way, or in a manner closely approximating thereto. The pier, No. 3, of Gault bricks in mortar, presented a good typical example. It bulged evenly on all sides at once, nearly every brick in the body of the pier was broken, and the two

First Series.

Brick.	Mortar used.	Age of pier. Weeks.	Pressure per sq. ft. of pier, in tons, at which the pier		Average.
			began to fail.	collapsed.	
London stock ..	Lime	18·3	4·18	10·41	10·41
Gault	"	18·3	5·00	21·82	
"	"	18·8	6·16	22·03	21·92
Leicester red.. ..	"	19·1	15·20	29·93	
"	"	18·6	16·11	31·55	30·74
Staffordshire blue..	"	18·6	22·43	69·22	
"	"	19·4	21·42	79·39	74·30
London stocks ..	Cement.	21·1	7·22	16·03	14·93
Gault "	"	21·0	5·72	13·83	
"	"	21·0	6·98	18·07	17·79
"	"	20·5	7·08	17·51	
Leicester red.. ..	"	22·1	17·87	67·36	58·45
"	"	21·7	21·82	49·54	
Staffordshire blue..	"	21·7	29·45	84·47	72·80
"	"	22·7	16·91	61·14	

pyramids at top and bottom, remaining after the crushing of the mass, were regularly defined and approximately concentric with the axis of the pier.

In one case the cement was found to be disintegrated, and as the corresponding pier, which was subjected to almost exactly the same pressure, showed its cement to be perfectly hard set, and uninjured by crushing, one is led to

the conjecture (says Mr. Garbutt) that the cement used in No. 12 had been left mixed overnight and beaten up again in the morning, or that the bricks had not been sufficiently wetted, and had withdrawn the moisture from the cement.

The results of the tests of the first series, which are set out in the reports in very full detail, may be shortly summarised as in the table on the preceding page.

The average strengths of the bricks used in building the above piers, as ascertained by Professor Unwin by his tests at the Central Technical College, are as follows:—

	Crushing strength in tons per square foot.
London stocks	84·27
Gault	182·2
Leicester red	382·1
Staffordshire blue	701·1

The results of similar tests of lime and cement mortar made in the proportions used gave the following:—

Lime Briquettes.

Strength at 4 weeks	6·08 tons per sq. ft.
" " 12 " 	8·73 " "
" " 24 " 	15·72 " "

Cement Briquettes.

Made with same sand as that used in building the piers. Strength at 24 weeks = 29·00 tons per sq. ft.

—————
Made with Leighton Buzzard sand.

Mean strength at 4 weeks	31·45 tons per sq. ft.
" " 13·7 " 	48·52 " "
" " 24·0 " 	56·15 " "

In order to ascertain the composition of the mortars used in the above experiments, a series of analyses were made in

the laboratory of the London County Council under my direction, at the request of the Committee, conveyed through Mr. Blashill, at that time superintending architect of the Council, with the results given in table on next page.

When reporting upon the second series of tests, the Committee observed that it was interesting to note the great difference between the strength of bricks and of brickwork, the enormous disproportion being due to the quality of the material interwoven with the bricks as a means of uniting and holding the mass together. The effect upon different kinds of bricks was variable, owing to some bricks being more susceptible to adhesion than others. Again, if the mortar be imperfectly mixed, or the joints and beds of the brickwork imperfectly filled up, the injurious effect on strength is incalculable. It introduces unequal pressures in different parts of the body, and the work fails in detail, until so much of the whole is destroyed that the remainder suddenly collapses.

The Committee laid much stress upon the care required in the selection, preparation, and application of the mortar. They observed, however, "that there is one comforting reflection, namely, that if a building is likely to collapse from want of strength, it is more likely to do so while the mortar is fresh and green than at a later date; that is to say, if the mortar once gets set and matured under its load, the building will live its life, a longer or a shorter one, according to the character of the building"—or, shall one say, of the builders? A few weeks ago, observes the Committee, in taking down some buildings less than a hundred years old, the bricks could be lifted piecemeal by the hand, and an overturn of 3ft. or 4ft. of the walls was sure to dislocate the whole of the bricks. On the other hand, in some buildings several centuries old, the mortar joints are as hard

First and Second Series.

Brick.	Mortar used.	Age of pier.	Pressure per sq. ft. in tons at which the pier collapsed.	Average.
Stocks	Lime	3 to 4 months	10'41	14'34
"	"	"	*18'28	
"	"	10 months	14'11	12'54
"	"	"	10'97	
Gault	"	3 to 4 months	21'82	21'92
"	"	"	22'03	
"	"	10 months	22'93	21'60
"	"	"	20'27	
Leicester red.. ..	"	3 to 4 months	29'93	30'74
"	"	"	31'55	
"	"	10 months	34'35	34'12
"	"	"	33'89	
Staffordshire blue..	"	3 to 4 months	69'22	74'30
"	"	"	79'39	
"	"	10 months	73'56	73'66
"	"	"	73'76	
Stocks	Cement	3 to 4 months	16'03	14'93
"	"	"	13'83	
"	"	10 months	22'34	19'65
"	"	"	16'96	
Gault	"	3 to 4 months	18'07	33'68
"	"	"	17'51	
"	"	"	*48'10	29'98
"	"	10 months	*51'05	
"	"	"	26'33	67'76
"	"	"	33'64	
Leicester red.. ..	"	3 to 4 months	67'36	67'76
"	"	"	49'54	
"	"	"	*86'39	50'43
"	"	10 months	47'86	
"	"	"	53'00	87'94
Staffordshire blue..	"	3 to 4 months	84'47	
"	"	"	61'14	87'94
"	"	"	*108'25	
"	"	"	*97'90	82'48
"	"	10 months	91'80	
"	"	"	73'17	15'0
Leicester red.. ..	In sand only	—	15'0	

* The piers were built in a manner very superior to the first two series, and therefore cannot fairly be averaged with them.

“as iron,” the joints protruding beyond the masonry, the surface of which had fretted away by the action of the atmosphere during the ages which had elapsed since the structure was built.

The results of the first and second series of tests are given in the table on page 141.

It will be readily noticed, observes the Committee, in looking down the columns, that something must have greatly disturbed these averages, as it would, for instance, be absurd to expect some of the descriptions to be weaker at ten months than at four. The cause, however, is not far to seek. The piers built as substitutes for those which the Committee found badly built in the first series, were so superior to any of those reserved for the ten months' test, that they raised the average for four months to beyond that obtained for the piers crushed at ten months. Taking, therefore, the original piers as specimens of what might be got in ordinary practice, one ought, perhaps, to reduce the averages to approximate to those obtained in the first instance from indifferent, which is indeed, the ordinary manner of building.

In the third report the Committee gave the results of experiments conducted to ascertain the average strength of various descriptions of brickwork by crushing twenty short lengths of brick walls, each about 6ft. high by 27in. long and 18in. thick. The results are set out, as before, in condensed tabular form on page 143.

Pending a more careful examination of the facts, the Committee derived the following impressions. The resistance of brickwork in lime mortar to crushing would seem to vary from one-sixth to one-eighth of the resistance offered by the brick itself, while in cement mortar it varies from one-half to one-fifth of that strength. It is obvious that while

cement mortar must very materially aid the weaker bricks in their combined strength, it cannot materially affect the ultimate power of resistance in brickwork made of a harder variety.

The average thickness of the bricks was $2\frac{1}{4}$ in., and the

Third Series—Brick Walls.

Brick.	Mortar used.	Age of wall.	Pressure per sq. ft. of wall in tons at which wall collapsed.	Average.
Stock	Lime	22 weeks	17'44	18'63
"	"	"	19'83	
Gault	"	"	31'34	31'14
"	"	"	30'94	
Fletton	"	"	30'82	30'68
"	"	"	30'54	
Leicester red.. .. .	"	"	45'94	45'36
"	"	"	44'78	
Staffordshire blue..	"	"	118'12	114'34
"	"	"	110'56	
Stocks	Cement	21 weeks	39'24	39'29
"	"	22 weeks	39'34	
Gault	"	"	51'50	51'34
"	"	"	51'19	
Fletton	"	21 weeks	54'88	56'25
"	"	"	57'63	
Leicester red.. .. .	"	"	80'94	83'36
"	"	"	85'78	
Staffordshire blue..	"	"	139'52	135'43
"	"	"	131'35	

total thickness of the mortar beds was 6 in., while the compression of the lime mortar beds averaged 1 in., and that of the cement mortar beds about $\frac{1}{3}$ in. This proves that the mortar generally was well crushed, and disintegrated long before the final collapse of the several examples of brick-

work. The instantaneous photographs taken by the Committee showed the mortar flowing out as in a stream or fountain at the moment of collapse.

In dealing with the working load that may be calculated upon, care must be taken not to impose such a load as would materially damage the structure of the brickwork. At one-fifth of the crushing load the compression in lime mortar averaged $\frac{7}{32}$ in. in 6ft. of brickwork, and in cement mortar it averaged $\frac{5}{32}$ in.

Another thing that will have to be remembered is the great difference between dead and live loads.

If, says the Committee, we take the safe load, or one that would not materially damage the structure, as one-fifth of the crushing load, it may be assumed from the results obtained, that in lime mortar 1 to 2, stock brickwork is equal to about $3\frac{1}{2}$ tons; Gault, 6 tons; Fletton, 6 tons; Leicester red, 9 tons; and Staffordshire blue, 23 tons per square foot. In Portland cement mortar, 1 to 4 stocks would be equal to about 8 tons; Gault, 10 tons; Fletton, 11 tons; Leicester red, 17 tons; and Staffordshire blue, 24 tons per square foot. This, however, is put forth as only a general assumption, which requires further consideration.

CHAPTER XII.

CONCRETE.

Definition.—Concrete is a solid mass formed from lime or cement, with sand, and small, irregular pieces of stone, brick, slag, clinker, &c., by their amalgamation together when brought into contact with each other by the aid of water. The amalgamative agents, or more properly speaking, the solidifying agents, are the lime or cement and the water; the other ingredients are inactive, being merely bound together by the foregoing. The two classes of ingredients which go to form concrete are technically known as the *matrix* and *aggregate*, *i.e.*, the *active* (cement and water) and the *inactive* agent (sand) respectively.

Béton.—Concrete is sometimes called by the French name of Béton. Between these two names there is a distinction without a difference, for both signify “concrete” as the word is understood in its wide sense. The distinction, however, lies solely in the operation of mixing the ingredients as practised in England and France. In this country the general practice is to mix the whole of the ingredients *dry*, and when they are thoroughly mixed water is added gradually, taking care, however, to turn over the ingredients during the addition of water. In France it is customary, first, to form a mortar by mixing the lime or cement with the sand and water; the bricks, stone, or other material being added to the mortar, thus formed, afterwards.

Historical.—If, in considering the history of concrete, we include that of mortar and cement, we must go back very

far indeed to arrive at the time when this substance in one or more of its various modifications was first employed in building construction. In the opinion of some authorities, the pyramid of Cheops is an excellent example of how little we have advanced in this respect on the knowledge of the ancient Egyptians; who, when they required the early Israelites to make "bricks without straw," evidently wanted a crude sun-dried "mortar" or "concrete" in blocks. It can hardly be contended, however, that these early bricks come under the head of mortar or concrete, but yet they point to a means of preparing earthy matters for building purposes, and doubtless the introduction of ashes and, later, lime, to the clay and sand, gradually developed the present system of making concrete as we now know it, with Portland cement and ballast. In Prescott's "Conquest of Mexico," stucco is frequently referred to, as also, "the sensations of the Aztecs as they heard for the first time, the *well-cemented* pavement ring under the iron tramp of the horses." It is certain that the Etruscans and Greeks imparted their knowledge in this respect to the Romans, who employed concrete in the construction of Corfe Castle in Dorsetshire, &c.

The use of Roman cement may be seen in various ancient buildings at Pevensey and Richmond, in the Norman and early English foundations of Ely Cathedral, in Salisbury Cathedral, Westminster Abbey, Guildford Castle, Middleham Castle (Yorkshire), and many other old English castles. An inspection of the ruins of feudal fortifications reveals the fact that concrete was very commonly used in the Middle Ages. The Castle of Badajos, in Spain, still bears the marks of the boarded frames in which the concrete was deposited. To the Chinese we must give credit for having been, if not the first, amongst the earliest nations who used concrete. The Great Wall of China, begun

in 214 B.C., was built mainly of concrete, but a "concrete" different to both our own method of mixing the ingredients and that of the French method.

Chinese Method of Concrete Making.—The Chinese method of preparing concrete for wall building is as follows:—The ingredients are 1 part lime and 2 parts of a mixture of either sand, gravel, and shingle, or sand, building *débris*, and earth. These ingredients are mixed thoroughly together *dry*. The wall intended to be built is first outlined by a wooden shell, or rather by boarded frames, and filled up by degrees with the dry concrete mixture described above. A layer of this mixture, about 6in. deep, having been put into the casing or shell of the intended wall, the workmen jump into the casing and begin to press and pound the mixture down by means of rammers—wooden poles having a base of about 6in. to 8in. diameter, tapering towards the top. The concrete mixture is by this means compressed to a sufficient compactness. Over this punned or rammed surface water is sprinkled, by means of a wooden bowl dexterously used by the foremen, whereby the surface is moistened, but not wetted; then some more of the loose concrete mixture is put into the casing, and the processes are repeated until the intended height of the wall is reached. A wall built in this manner divides the Portuguese city of Macao, China, from the Chinese territory of Heong-San. It has stood for nearly 250 years, and my informant, Mr. Francombe, who has lately seen it, says, "It is still in splendid condition."

The Romans were great concrete builders. They made use of it before the year 500 B.C. Vitruvius, who wrote *circa* 100 B.C., gives instructions for its manufacture. He also describes the process of making concrete for pavements and floors, and concrete walls around wells.

His specification for the concrete in the last instance is as follows (according to Gwilt's translation):—"In the first place, the purest and roughest sand that can be had is to be procured. Then material is to be prepared of broken flint, whereof no single piece is to weigh more than 1 lb. The lime must be very strong, and in making it into mortar five parts of sand are to be added to two of lime. The flint work is combined with the mortar, and of it the walls in the excavation are brought up from the bottom, and shaped (Professor Aitchison, A.R.A., renders the word "rammed" instead of "shaped") by wooden bars covered with iron."

Alberti and Palladio, in the fifteenth and sixteenth centuries, described methods of building concrete walls. Amongst the works of note constructed in more recent dates wherein concrete was used may be mentioned the Millbank Penitentiary (in 1811), by Sir Robert Smirke; the Graving Dock and Sea Wall at Woolwich (1835), by Ranger.

Concrete in Building Construction.—The employment of concrete in building construction has received a great impetus since the introduction of Portland cement—undoubtedly the strongest cement the world has ever known—which has on account of its great strength and reliability led to the use of concrete for many purposes for which lime-concrete was utterly unsuitable, with the result that concrete is now used in foundations, floors, walls, roofs, drain pipes, stairs, conduits, arches, pavings, bridges, lintels, building-blocks, and has even been employed in making doors, telegraph poles, and in the erection of mill chimneys, either alone or in combination with wood, iron, steel, &c. With the advancement made in the use of cement-concrete, numerous patents have been taken out for improvements

both in the methods of making and using this substance, some of varying merits, while others have been failures involving great pecuniary losses to the inventors ; and to-day concrete, notwithstanding many failures—the stepping-stones to success or advancement—is used for a larger variety of purposes than ever before.

Mixing.—The nature of limes and cements for specific concrete works have been dealt with in a previous chapter. In the preparation of concrete a great deal depends upon how the matrix is mixed with the aggregate ; its importance has been recognised by the early users of concrete, with the result that various concrete-mixing machines have been invented—some made in sizes small enough to be worked by hand ; but those driven by steam power are the ones mostly in general use. Concrete is more thoroughly and rapidly mixed by machinery than by hand, and for considerable quantities it is much cheaper, apart from the fact that machines do their work with more uniformity than labourers, and constant supervision, so necessary when the latter is employed, may be lessened. Many architects are now specifying that all concrete in large quantities shall be mixed by machinery. Care will, however, have to be experienced by them in the choice and approval of concrete-mixing machines, as many of these seem designed rather to turn out a large quantity of concrete than to ensure the thorough mixing of the materials. The thorough incorporation of the matrix with the aggregate should be the main point to be considered in the selection of these machines.

Depositing.—As soon as concrete is mixed, no time should be lost in depositing it, as any disturbance of the mass after the cement or lime has begun to set detracts from the ultimate strength ; more special attention in this regard should be given where quick-setting cement is

Concrete Block: Compressed.

Portland cement concrete blocks of various materials, set and kept in air for one year; also set and kept in water for the same time. Materials, various; size of block, 6in. by 6in. by 6in.; moulded, November 4th, 1867; tested, November 4th, 1868.

Proportion.	Weight in pounds.			Weight of each block in pounds.		Crushed at tons.		Remarks.
	Cement.	Sand.	Water.	Kept in air.	Kept in water.	Air.	Water.	
6 to 1	2·26	15·31	1·00	17·90	18·60	20·40	19·60	Ballast
	2·40	12·80	1·45	16·90	17·67	40·60	34·50	{ Portland stone
	2·31	14·56	1·30	18·53	18·88	30·50	27·0	Granite
	2·16	13·33	1·75	16·10	17·50	28·80	26·50	Pottery
	2·11	12·29	1·60	15·08	16·61	23·0	23·50	Slag
	2·03	13·91	1·45	16·50	17·65	20·50	24·0	Flints
	2·37	15·51	1·30	18·50	19·25	28·0	23·0	Glass
8 to 1	1·76	15·88	·95	17·86	18·90	13·50	13·50	Ballast
	1·86	13·26	1·65	16·32	17·50	33·00	29·00	{ Portland stone
	1·53	14·57	1·25	18·10	19·0	19·60	16·00	Granite
	1·64	13·53	1·60	16·15	16·95	22·00	23·00	Pottery
	1·60	12·37	1·50	14·20	15·91	19·50	13·40	Slag
	1·56	14·28	1·30	16·45	17·62	17·50	20·00	Flints
	1·82	15·79	1·15	18·00	18·93	18·00	17·50	Glass
10 to 1	1·42	16·11	·85	17·68	18·70	10·50	10·50	Ballast
	1·52	13·56	1·86	16·44	17·90	22·00	16·50	{ Portland stone
	1·23	14·60	1·15	17·60	18·50	15·50	12·40	Granite
	1·34	13·75	1·45	16·09	16·80	18·50	19·00	Pottery
	1·28	12·46	1·40	14·00	15·50	10·50	10·00	Slag
	1·26	14·37	1·15	16·15	17·60	15·00	18·50	Flints
	1·48	16·16	1·05	17·90	18·73	12·50	12·80	Glass

Concrete Block : Not Compressed.

Proportion.	Weight in pounds.			Weight of each block in pounds.		Crushed at tons.		Remarks.
	Cement.	Sand.	Water.	Kept in air.	Kept in water.	Air.	Water.	
6 to 1	2'00	13'49	'88	17'35	18'30	18'20	17'00	Ballast
	2'23	11'91	1'35	15'70	17'12	30'00	23'00	(Portland stone
	1'98	12'55	1'12	17'40	18'30	24'50	15'50	Granite
	1'91	11'78	1'54	15'85	17'00	24'60	24'00	Pottery
	1'95	11'36	1'23	14'80	15'77	20'00	19'20	Slag
	1'76	12'11	1'26	15'20	16'60	15'50	15'50	Flints
	2'04	13'35	1'11	17'76	18'40	16'50	17'00	Glass
8 to 1	1'60	14'51	'85	17'38	17'95	12'50	11'00	Ballast
	1'70	12'15	1'51	15'63	17'00	24'50	19'50	(Portland stone
	1'31	12'53	1'07	17'45	18'20	14'50	13'40	Granite
	1'43	11'83	1'40	15'71	16'70	18'00	18'00	Pottery
	1'47	11'37	1'37	13'73	15'12	14'00	9'50	Slag
	1'34	12'02	1'12	15'40	16'60	14'00	12'50	Flints
	1'52	13'22	'96	17'30	17'80	13'60	11'40	Glass
10 to 1	1'26	14'35	'75	17'20	17'50	8'00	7'00	Ballast
	1'38	12'34	1'69	16'15	17'00	19'00	10'00	(Portland stone
	1'05	12'62	'99	16'97	17'63	11'50	11'00	Granite
	1'15	11'88	1'25	15'66	16'50	14'00	17'00	Pottery
	1'17	11'43	1'28	13'75	15'00	8'50	5'50	Slag
	1'13	12'00	'90	15'12	16'60	12'80	11'00	Flints
	1'22	13'31	'86	16'95	17'70	10'00	10'80	Glass

employed. When the concrete is in large masses, as in harbour works, caissons, retaining walls, foundations, &c., it is customary to use *packing*, that is to say, large pieces of stone are inserted for the sake of economy. This can safely be done provided that all *packing stones* are thoroughly wetted before being laid in the work, and that they are not too near each other; care should be taken that there is a sufficient excess of mortar in order that the *packing* may be properly united with the rest of the mass.

Ramming. — Compression increases the strength of concrete, as also its density, and, in consequence, its imperviousness to water and its durability. Without ramming it is impossible to have impervious concrete. On the other hand, if ramming is resorted to in large masses of concrete, there is the risk of interrupting the process of setting, which commences immediately on the application of moisture. An increased proportion of cement should be added if it is desired to augment the strength of the concrete. Ramming, however, may be safely recommended in making concrete bricks or blocks of moderate size, states my former colleague, the late Mr. John Grant, who carried out a series of experiments to prove this. In his work on the "Strength of Cement," page 140, a series of tables are given, in which he shows the results of his experiments, abstracts from which are reproduced on pages 150 and 151.

The compression was effected for the above experiments by beating the concrete into the moulds with a small mallet. As will be seen by the table given above, the average gain in strength was—

30 per cent.	for 6 to 1 mixture kept in air,
37½	„ 6 to 1 „ „ water,

27 $\frac{3}{4}$	per cent.	for 8 to 1	mixture kept in air,
39	„	8 to 1	„ „ water, and
24	„	10 to 1	„ „ air,
44	„	10 to 1	„ „ water.

This is a gain that commends itself to builders and to manufacturers of concrete drain-pipes and artificial stones, as the latter almost invariably compress the raw material in one way or another, in order that the goods may be strong and impervious to water. As concrete very frequently has Portland cement as a matrix, it requires protection not only against traffic, but also against extremes of temperature and atmospheric influences, which should be avoided as much as possible. Mr. W. W. Mackay, of the Docks Department, New York, found in 1876 that Portland cement briquettes made and kept in water at a uniform temperature of 60 deg. Fah., were at the end of seven days 30 per cent. stronger than exactly similar briquettes which were made at a temperature of 60 deg. to 70 deg., and kept in water varying daily from 70 deg. down to about 40 deg. Again, Mr. Fitzmaurice gives the results of his experiments in Nova Scotia, during the winter of 1890-1:—"Exposure to natural frost for four days—out of a total of twenty-eight days—reduced the strength of neat Portland cement briquettes 15 per cent., and of cement and sand briquettes (1 to 3) from 28 to 35 per cent. Exposure for the full period of twenty-eight days reduced the strength of the former 35 per cent., and of the latter no less than 57 per cent." Practical architects maintain that concrete setting at ordinary temperatures is of satisfactory strength, while it is considerably weakened by frost, and that in extreme cases it is rendered worthless.

Hastening the Setting of Concrete.—The matrix for making

such a concrete should be composed of the best quick-setting Portland cement used fresh, and less sand should be used, taking care to reject fine sand and dust, as these greatly retard that action; and last, but not the least, no more water should be used than that which is absolutely necessary. If a lesser quantity of aggregate be incorporated with this matrix than is usual, the rapidity of the setting of the concrete would be hastened; and it would be further accelerated if the temperature of its surroundings was likewise increased by steaming. Moist heat, as already pointed out, hastens the setting of concrete. The surface of the concrete so laid may be further protected by covering it with non-conductors of heat, such as straw, hay, bags of sawdust, &c.

When concrete is well prepared, even in the ordinary way, it is capable of supporting the action of the London atmosphere successfully, as well as the action of water.

The great point to be observed is to properly appor-tion and to intimately mix the lime or cement into the state of mortar, and then to present it to the materials round which it is intended to crystallise in such a manner that equality of setting may be secured. Well selected blue lias lime, or grey stone lime, mixed with pounded brick dust, will do well for the execution of concrete for the ordinary conditions of exposure to the weather. But for exposure in sea-water the employment of Portland cement is necessary.

Mr. E. Puscher, according to the *Journal* of the Chemical Society, March, 1883, recommends the following process for rendering cement and lime less subject to atmospheric influences:—The cement materials should be allowed to remain in a cold-water solution of one part ferrous sulphate

in three parts of water for twenty-four hours, after which they are dried in the air. The ferric oxide produced is mechanically combined in the cement, and makes it denser, harder, heavier, and weatherproof, filling up most of the pores, and giving it an ochre colour. Ornamental cement work is brushed over with the solution four times and allowed to dry. The cement work can be rendered extremely resisting by warming and then coating with a hot mixture of equal parts of paraffin wax in light petroleum. By treating twice with a 5 per cent. soap solution, drying and polishing, the surface is made efficient for oil painting. Chalk objects and room walls treated in this manner will stand any amount of washing. Light ochre colour can be obtained by adding alum to the ferrous sulphate; and various shades of green by painting with chrome-alum.

Hydraulic silica, gelatinous silica, and soluble silica absorb lime gradually from lime water, the maximum absorption varying in all cases, according to Mr. E. Laudrin,* between 36 and 38 parts of lime for one equivalent of silica. The resulting compound has approximately the composition of $3 \text{ SiO}_2, 4 \text{ CaO}$. The combination is most rapid in the case of soluble silica, but even in this case the maximum absorption is not effected until after several hours. Silica from hydrofluosilicic acid absorbs lime much more slowly than any of the three previously mentioned varieties. The maximum absorption after sixty-eight days, in a series of experiments, was 24.2 parts of lime per equivalent of silica. For this compound ($3 \text{ SiO}_2, 4 \text{ CaO}$) Mr. Laudrin proposes the name Pouzzo-Portland. When mixtures of lime with different varieties of silica in the proportion required to form Pouzzo-Portland are heated to

* *Journal of the Chemical Society*, August, 1883.

bright redness in a gas-carbon crucible for a time, varying with the nature of the silica, the fused, but non-vitrified mass yields an artificial Pouzzo-Portland which generally splits up and falls to powder as it cools. It is completely soluble in hydrochloric acid, and when moistened with the smallest possible quantity of water, and immersed under water, it sets in from fifteen to sixteen hours, acquiring a hardness, which, however, is scarcely equal to that of Spanish white. If, however, the water is charged with carbonic acid, the cement after some hours acquires a hardness equal to that of the hardest stone. This fact tends to show that the absorption of carbonic anhydride is an important factor in the setting of hydraulic cement.

Specification for Concrete.—There are numerous specifications for concrete, and it is impossible to lay down any definite hard-and-fast rule, as the mixture of the ingredients will necessarily vary according to the requirements of the case and the experience of the engineer. The following are given as indications only which may be useful to the reader.

Without specifying the use to which it is to be put, Newbigging prescribes 1 part of lime, 4 parts gravel, and 2 parts sand.*

On the other hand, Mr. Boulnois gives the following † :—

“Street Foundations—Heavy Traffic.—

Portland cement	1 part.
Sharp river sand	2 „
River ballast or broken stones ...	4 „

* Newbigging's "Handbook for Gas Engineers."

† "The Municipal and Sanitary Engineer's Handbook." Boulnois. Page 100, &c.

“*Liverpool Street Foundations.*—

Portland cement	1 part.
Gravel	5 to 6 parts.
Broken stone	7 to 8 „

The gravel and cement are thoroughly mixed dry, and only enough water then allowed to flow on it to make the material damp enough after it is incorporated to retain its form when a portion is taken in the hand and squeezed.*

“The ground having been excavated, thoroughly consolidated, and properly graded to the requisite shape, a layer of broken stone, or other material, is spread evenly over the surface and thoroughly wetted from the rose of a watering can. A stratum of concrete, mixed as above, is spread over this, and a second layer of stone added. The stone is then beaten in with a heavy flat beater. Other layers of mortar and stones are added and thoroughly beaten in until the required thickness is obtained, the final layer of cement concrete being smoothed off to an even and uniform surface. After this concrete, or *béton* foundation has been allowed to set ten days, the paving is commenced.”

For footpaths, Mr. Boulnois recommends a concrete made with 1 part Portland cement, 2 parts clean coarse gravel, passed through 1in. mesh, and 2 parts of clean sharp sand. A finishing coat may be composed of 1 part Portland cement, 2 parts granite chippings.

* This, it will be seen, is the Chinese method.

CHAPTER XIII.

ARTIFICIAL STONE.

History.—To what era the introduction of artificial stone belongs, it is not within the scope of this chapter to discuss. Its antiquity, however, has been established by the archaeological researches carried out in Egypt, which have clearly shown that at least one of the Pyramids was constructed of artificial stone, *i.e.*, concrete blocks made of small round stones, broken stone, and lime.

In many of the old remains, still existing in England from the periods of Roman occupation, can be distinctly traced blocks of various shapes and sizes made from a concrete in which flint played a prominent part.

Coming to more recent productions, at the end of the last century (eighteenth), a French architect carefully worked out a process for making blocks of stone with a special matrix, *i.e.* cementation principle, of cement which he made with lime, clay and charcoal.

Later, in England, just before the Victorian era, a Mr. Ranger took out several patents for the manufacture of a concrete which was used in the walls of the Woolwich Dockyard Wharf. Following this we have Coignet's "béton aggloméré," a composition of sand and hydraulic lime or cement, and sometimes both. This was largely used in France, but met with little favour in this country. Besides sewers and numerous smaller works, blocks of it were used for the Suez Canal, and a church at Vesinet, near Paris, having a steeple 135ft. high, was erected of it.

Ransomes' "Siliceous Stone" was perhaps the first

artificial stone which was prepared on a scientific basis. Washed sand (1 bushel) and a solution of silicate of soda, specific gravity 1.7 (1 gallon), were mixed in a pug-mill until the whole was thoroughly incorporated and was of the consistency of putty, when it was moulded into blocks or slabs, as required, and drenched with a solution of chloride of calcium, then placed in tanks containing the same solution and heated to boiling. The resulting stone, the hardness of which depended on the formation of calcium silicate as a matrix, met with considerable success; but there was connected with it one unfortunate difficulty. It was necessary for it to be prepared with extreme care—a care which only skilled labour was capable of and, as a result, the process was found to be more or less commercially impracticable and fell through.

Buckwell a few years later introduced a material which he called "Granitic-Breccia" stone, which was composed of Portland cement and oolitic or magnesium limestone, and made by coarsely crushing the limestone, incorporating it with the cement with the least possible quantity of water, and ramming it into moulds. Owing to the retirement of Buckwell, however, the operations fell through, but a stone somewhat on the same principle is still made in considerable quantity, mention of which will be found later.

These two stones, Ransomes' and Buckwell's, have been mentioned at some length, as they are practically the forerunners of all the modern artificial stones. Of these probably the best known is—

The Victoria Stone.—This stone was introduced by Mr. Highton many years ago, but, handicapped at that time by the variable quality of Portland cement, due to its unscientific manufacture, and owing to experimental errors with unsatisfactory materials, it did not at once meet with

the success the material really deserved. About this time, however, came a revolution in the manufacture of cement. The Germans, studying the manufacture of Portland cement from a scientific point of view, succeeded in making a cement of practically constant composition and value, and thus temporarily almost ousted their English rivals from the market. As a result of this, the English makers, finding their trade steadily declining, called in the best scientific talent to assist them, and by the aid of combined research succeeded in producing a cement which has practically no equal.

With the use of a cement of practically constant composition, and a granite of exceptional hardness, the stone rapidly rose in favour, and for over twenty-five years has now held a prominent position.

This stone, which for many years was used almost entirely for the making of paving stones, is a good example of what can be done in concrete by the careful treatment of suitable materials.

It is manufactured from a granite obtained from Groby, in Leicestershire, the composition of which is :—

Silica (insoluble)	65·26
Silica (soluble)	0·55
Alumina	13·06
Lime	4·55
Magnesia	1·01
Oxide of Iron	9·81
Soda	2·34
Potash	2·85
Carbonic Acid	0·03
Water, &c.	0·54
	100·00

The hardness of this granite is even greater than Guernsey granite, its crushing strain being about 20,750 lb. per

square inch. The granite is first finely crushed, and then washed as free as possible from alkalis and alkaline salts,* and mixed in suitable proportions with Portland cement—generally about 3 parts of granite to 1 part cement—in the dry state by machinery, water is then carefully added, care being taken to prevent elutriation, and whilst still plastic it is placed in moulds, where, by shaking, pugging and the judicious use of a trowel, a solid block is obtained free from air-bells, and in such a state that the process of induration which it next undergoes will have the maximum effect. When the concrete blocks thus formed have sufficiently hardened, the moulds or frames, which are made of wood lined with metal and fastened at the ends, are removed and the blocks treated with a solution of alkaline silicates in a bath for varying periods, generally about fourteen days, after which they are exposed to the atmosphere for some time to attain their maximum hardness—a hardness, of course, due to the combination of the silicate with the free lime of the cement.

The stone thus produced has a chemical composition of:—

Silica	50·35
Alumina	11·87
Oxide of Iron	7·33
Lime.. .. .	18·33
Magnesia	2·03
Potash	1·78
Soda	3·81
Carbonic Acid.. .. .	1·80
Water, &c.	2·70

100·00

* Too much stress cannot be laid on the freedom of all stones from alkaline salts, especially the sulphates of the alkalis. The crystallisation and solution of these salts due to atmospheric con-

a crushing strain of about 8300 lb. per square inch, about 30 per cent. more than Peterhead granite, a breaking strain of about 1300 lb. per square inch, and a porosity of 1·3 per cent. on a 24 hours' absorption test, being one-tenth that of Portland stone, one-eighth that of Mansfield stone, and practically equal to that of marble. The results obtained by mechanical tests such as these give a very good idea of the quality of a stone, still a test of actual wear is worth recording.

In Islington, outside the Agricultural Hall, where the traffic probably is nearly as heavy as any place in the metropolis, this stone, after being laid for thirteen years, had to be removed owing to alterations to the footway, &c., but finding that it was in such good condition the local authorities decided, on completion of the alterations, to re-lay it on the reverse side, so that the new additions and the old should be comparable. This was done some eight years ago and the stone is in as good condition as ever.

As has been mentioned before, this stone was for some time used only for paving, steps, window-sills, &c. ; of late, however, considerable attention has been paid to the production of a building material which should rival all ordinary natural stones. This is made in identically the same manner as the stone for paving, except that the size of the crushed granite is slightly varied to suit the circumstances, and that it is, if required, coloured by the addition of either the yellow or red oxide of iron, to imitate the yellow or red Mansfield stone respective y.

This introduction has met with a marked success—a success which is probably due as much to the way the

ditions cannot fail to finally cause disintegration of any material containing them to any degree.

material is handled as to the material itself. Its extreme hardness and closeness of grain renders it a most suitable material for carving, the sharpness of the lines and contour being all that could be wished. For this purpose the blocks are cast to the approximate shape required, the whole of the carving being done with the chisel, as in ordinary stone.

One important fact in connection with this stone is the little effect weather and the atmosphere, even of manufacturing towns has on it. None, perhaps, have recognised the defects of natural stone more thoroughly than those who have been responsible for architectural work, or had to build, near the sea. The deplorable spectacle of clock-towers, memorials, and hotel fronts pitted and weather-eaten is familiar to all—a condition produced chiefly by the crystallisation of the salt within the pores of the stone, and the consequent flaking, &c. The low porosity and hardness of this patent stone enables it to resist the action of even the sea air—a fact which is now beginning to be recognised and taken advantage of, the substitution of artificial for natural stones being noticeable all along our coasts. Amongst others the Terriss Memorial at Eastbourne, and Morley House Convalescent Home at St. Margaret's Bay, the entrance to which is an excellent example of what can be done in architectural reproduction, is built of this stone, and has fully justified the opinion of those who advocate the advantages of artificial over natural stone.

One cannot but look favourably on a stone such as this, which, less costly than even the soft Bath stones from which all detail is effaced in a few years, free from the too frequent flaws in natural stone, flaws which unfortunately only show up after some little wear, ready of production

and constant of composition and durability, is made on a sound scientific basis.

Ward's Stone.—Under this name are two distinct stones. The first, which calls for but slight mention, is made from crushed granite and Portland cement, somewhat on the principle of the "Victoria Stone," except that no process of induration is used, the desired effect being claimed to be obtained by using a special cement which prevents air cracks, &c., in drying. This stone is largely used for paving, stable flooring, &c.

The other stone has been mentioned before in the opening part of this chapter. It is practically a modernised modification of Buckwell's "Granitic Breccia" stone.

For the manufacture of this material various limestones, chiefly of the oolitic variety, are coarsely crushed, mixed with a special Portland cement, and cast in moulds or frames, according to the block required. When thoroughly set and hardened, the surface is rubbed down to whatever design is required and finally polished. Slabs or mouldings in any form, or anything which can be cast to the approximate size and form required are suited to it, but it is not suitable for carving on account of the varying hardness of the different particles of limestone, &c., set in the matrix.

In its finished state the stone bears a marked resemblance to, and, in fact, might be considered as an imitation Belgian stone, it takes a remarkably good polish, considering the nature of the material, yet is not and does not become slippery. Owing to its decorative properties, combined with its durability, it has been largely used for ornamental staircases, the steps, &c., to the chancel of the new Roman Catholic Cathedral at Westminster being amongst the many uses to which it has recently been put.

Granolithic Stone—Stuart's.—This is a noteworthy example of the granite and cement-matrix stone. Particular attention has been paid to the production of a stone capable of resisting the continuous action of water, and to its adaptation to fireproof work—two extremes which speak well for the material. Probably no more severe test could be applied than intermittent submersion, a test which only picked natural stone will withstand. The successful construction of docks, such as the Glasgow graving dock, and water terraces is, perhaps, the best answer that could be given to critics who seek to disparage artificial stone.

“Non-slip” Stone.—For many years hard York stone was regarded as the standard material for paving, &c.; but, owing to the laminations which are inseparable from it, together with its irregular size and thickness and variable durability, it came to be looked on as far from an ideal standard. The constant trouble caused by this material to some extent prepared the way for the introduction of an artificial stone which could be obtained in regular sizes, even thickness, and square edges throughout the full thickness.

Recognising that it was impossible to obtain a surface which did not become more or less polished, and therefore not have the foothold of York stone, when granite was used as the base of the artificial stone, one of the large York stone companies took out a patent for the manufacture of an artificial stone, using York stone chippings as its base.

This material, which has been named “non-slip” stone, is made by crushing hard York stone chippings, and intimately mixing them with a suitable cement, casting the mixture into moulds and submitting it to hydraulic pressure. By this means a stone is produced having a surface practi-

cally the same as York stone, and a greatly increased durability, besides the advantages already mentioned.

Moreau Marble.—In nearly all the artificial stones that have been introduced Portland cement forms an essential ingredient ; practically, therefore, they are concretes.

In addition to these concretes, artificial stones have from time to time been introduced, with more or less success, having for their basis the changing of unsuitable natural materials into hardened materials closely resembling natural stones.

Probably the most noteworthy of these is one to which the name “Moreau Marble” has been given, the formation of which, although cementation undoubtedly plays a part in it, does not depend on Portland cement for its cohesion.

This stone—the name marble is somewhat of a misnomer, although the finished article bears the closest resemblance to it—can be produced from practically any amorphous variety of limestone, provided it is sufficiently soft, varieties which are worthless for building purposes.

The limestones which, however, are usually used in the manufacture of this “marble” are of two varieties, both of which have great uniformity of structure and evenness of surface. One variety is obtained from the neighbourhood of Poitiers, and the other, a fossiliferous limestone, from Angoulême, in both of which places huge deposits occur.

The process of formation is simple, the limestone being first cut to the approximate size, and then planed down by means of a steel cutter to the moulding or surface required.

The requisite shape having thus been obtained, the stone is then veined by a method somewhat similar to that used in chromo-lithography, the veins which it is desired to reproduce in the stone being first of all rendered impervious by the use of a suitable varnish, which, soaking into the

stone, prevents that part from absorbing the colour which is next applied.

In order to produce more natural veining than it would be possible to obtain by hand, a solution of 'gum Thus' in turpentine, with the addition of a little insoluble colouring matter, is floated on the surface of water and spread by a spray of soapy water. The face of the stone is then placed on the surface of the water, and thus picks up the streaks and patches which afterwards become the veins, &c., of the finished stone.

The stone is next coloured by immersing it in one or more solutions of metallic salts for different periods according to the shades required, thus producing a coloured stone by the same material with which the natural marble is coloured, and finally, if necessary, the colour is fixed by hot water.

Having thus obtained the coloured and veined stone, it now only remains to harden the material, which is done by immersion in a solution of sulphate of zinc for a period—generally about 24 hours.

It is somewhat difficult to understand why sulphate of zinc should have such an effect; certain it is that practically the whole of the zinc introduced is converted to carbonate, yielding an equivalent quantity of calcium sulphate, and probably the zinc carbonate thus formed acts as a stopping, filling in the interstices in the loose molecular structure of the limestone, whilst the resulting calcium sulphate crystallising out slowly in the structure sets up a state of tension in the stone, which still further tends to harden it. However, though the action is uncertain, the result is highly satisfactory. The stone, after its hardening bath, is dried first in a chamber at about 122 deg. Fah. (50 deg. Cent.), and finally in a chamber at about 212 deg. Fah. (100 deg.

Cent.), when it is ready to receive its polish in the same way as ordinary marble.

When it is required to imitate Rouge Royal marble, or to produce a "marble" which it is known will withstand continued heating, the stone is coloured by salts of iron, and treated in the same way, except that after drying it is exposed to a heat of about 800 deg. Fah. (446 deg. Cent.) in an oven.

The stone thus produced, as mentioned before, is exceedingly like in appearance to an ordinary marble, and takes a polish quite equal to it.

Too much stress cannot be laid on this point, as the higher the initial polish on any carbonaceous stone the better able it is to withstand the attack of the sulphurous and sulphuric acids present in the atmosphere of every town.

It has a hardness closely approximating to marble, and a crushing strain of about 4100 lb. per square inch, the crushing strains of marbles varying from 2240 lb. to about 6000 lb. per square inch.

In carving, sharpness of detail, and effect, it certainly is equal to marble. In only one instance, probably, does it fall short of marble; the initial cream colour of the limestone proves an insurmountable difficulty, and renders it impossible to reproduce the white marbles used for sculpture, &c.

Over its compeer it certainly has several advantages. Slabs and columns—that in the foreground of the illustration is in one piece—which would be prohibitive in marble, could readily be made in this stone.

It is free from the objectionable "stopping" which, for economic reasons, is so prevalent in many marbles; it is much less costly, and can be produced in a comparatively

short space of time. In an ordinary way the stone can be prepared and the process completed in twenty-one days; that erected in the entrance hall of the Balham Theatre being actually completed and fixed in sixteen days.

Having regard to the possibilities of colour, design, &c., it is perhaps somewhat to be regretted that so much attention is paid to making close imitations of marble. With such illimitable advantages of schemes of colour, easiness of working, and size, it would certainly seem better for the material to be treated more on its own merits.

It certainly should not be considered an imitation marble; it does not possess marble's essential feature, crystallographic form. It is a stone standing by itself, possessing all marble's advantages and more, but it commences the process as a stone and ends as a stone.

Terra-cotta.—Any account of artificial stone would scarcely be complete without mention of what is probably the most enduring of building materials, namely, terra-cotta, although it cannot by any means be considered as an artificial stone any more than a brick can. Yet on account of its process of manufacture its inclusion needs no apology.

Dating from many centuries B.C. its peculiar applicability has only been fully recognised during the latter end of the nineteenth century, mainly owing to misconceptions which arose as to its mode of architectural treatment.

Spread over the surface of the world are huge deposits of clay more or less suitable for the manufacture of terra-cotta, amongst those particularly adapted for the purpose being the North Devon and Dorsetshire clays, which contain in small quantities the alkalies which are necessary to aid the vitrification of the surface when the material is fired, and having for their composition ingredients which render

them suitable for working, practically without admixture. These clays have the following composition *:—

	North Devon.	Dorsetshire.
Alumina	29·28	32·11
Silica	52·06	48·99
Lime	0·43	0·43
Magnesia	0·02	0·22
Oxide of Iron ..	2·37	2·34
Potash	2·29	2·31
Soda	2·56	2·33
Water of Combina- tion	10·27	9·60

The clays obtained from some of the coal measures are also found, when properly used, to be most suitable.

The principal makers of terra-cotta have found, however, that they obtain more satisfactory results by a judicious mixture of clays, &c., a process which, though more expensive than using the natural clay, they consider amply justified by the improved result. What probably gives the manufacturer most trouble in the production of this material is the shrinkage which takes place in drying and firing. In order to reduce the amount of shrinkage and to aid in the drying, it has been found desirable to add refractory materials, such as previously hard-burnt clay, to the raw clay and ground with it. The manufacture may briefly be summed up as follows:—

To clay which has been carefully mixed and brought up to a standard of known shrinkage by the addition of refractory materials is added a vitrifying agent, when, after again carefully mixing with water to the required degree of plasticity, the prepared clay is ready to be pressed into the moulds which give it the desired form, and then dried by hot air, and fired in a suitable kiln.

* Analyses by Weston.

In the use of terra-cotta several points must be kept in mind. The surface of this material being slightly vitrified is the most resistant, and therefore care must be taken to preserve it intact, *i.e.*, free from chips; it should be sufficiently porous at the joints to make a perfect bond with the mortar; great care should be taken that all exposed joints are made good with cement mortar—an absolute necessity with this material. Large columns and, therefore, classic architecture, are unsuited to the material; designs should not call for larger blocks than 2 cubic feet, and should generally not exceed 1ft. cube. Any detail can be reproduced, but nothing produces such effect as that which shows the hand of the modeller of the natural material—clay. One of the earliest examples of the use of the material for a large public building in modern times may be seen in the Natural History Museum at South Kensington. The architectural outlines as seen from a distance are particularly striking, whilst on closer inspection one is charmed by the wealth of detail.

For many years terra-cotta was kept back and nearly ruined by the specifications of architects, who, either from lack of artistic taste, or because their specifications had always been drawn in this way for stone, &c., demanded uniformity of colour.

No greater blow could have been aimed at this industry than such a demand. The struggle for uniformity of colour tended towards under-firing the material, with consequent weakness and perishability, at the same time robbing it of all its natural qualities of play of colour and evidence of its presence in the fire. In the same way architects, without studying the nature of the material they had to deal with, treated it as if it were an ordinary stone, and prescribed for it all sorts of impossible purposes, then, finding

it could not be made to conform to their ideas, proceeded to damn it.

With the increasing recognition of its value, however, a number of architects have, by making a special study of the subject, and by availing themselves of the help of experts in the employ of the larger manufacturers, succeeded in demonstrating many legitimate treatments of architectural subjects in this material, and even in some cases have produced beautiful buildings with characteristics which could hardly have been obtained in any other material. The delicate divergencies of colour have been expanded, and schemes of architecture suited to its peculiarities developed, with the result that buildings have grown up around us possessing artistic individualism and affording a pleasant relief to the gloomy repetition so prevalent in our great cities, for which reason, if for no other, the development of terra-cotta is to be welcomed.

The foregoing summary includes the most important artificial stones now in use. Besides these, from time to time a number of different stones have been introduced, having in common with most of those described a cement matrix with a varying base, such as crushed coke, slag, &c. During the last few years several patents have been taken out for the production of a stone on an entirely different basis to any of the above, being made from silica, in the form of fine sand, and lime only. Although varying in detail, the principle is the same, *i.e.*, the combination of the lime with the silica by superheated steam under several atmospheres pressure. By this means blocks consisting of silicate of lime, silica and lime, have been made possessing to all appearances the characteristics of natural stone, but in reality having many points in favour which natural stones never have or can have.

The stone produced by one of the most recent patents has, indeed, the peculiar defect that unless worked within a few days it becomes so hard that ordinary masons reject it as being useless to them, they being unable to deal with it with their ordinary tools. When, however, the details connected with its manufacture have been completed it will undoubtedly take a high standing as a building stone.

To anyone who has studied the subject of artificial stone it becomes a matter of wonder why, possessing the many advantages it does, it is not more generally used. I believe the real answer to this may be found in the general conservatism of the architectural profession; trained as they are to architectural styles and laws handed down from generation to generation, they naturally regard materials somewhat in the same light, and so, in copying classic monuments of architecture, they try to obtain stone as closely resembling the original as possible.

Their one reply to such a question is that they have always used natural stone, and they prefer it. No reason, no argument; they have to prescribe, and they prescribe what their grandfathers did before them.

Doubtless, however, some prejudice has arisen against artificial stone owing to the action of unscrupulous merchants, who have put on the market worthless rubbish, generally with a name somewhat similar to some well-known make. A very simple rule, however, eliminates any such possibility as this, and is therefore worth mentioning: insist on seeing how the material is made that you are going to use, and if it is a genuine stone every facility will be granted you; if a fraud you will be put off with garbled information.

Year by year artificial stone is gaining in popularity, and studying its possibilities, one can scarcely doubt its future.

CHAPTER XIV

ASPHALT.

By the kind permission of Mr. Clifford Richardson, of Long Island City, New York, I am enabled to present to my readers the following most interesting and valuable account of the nature and origin of asphalt. These researches are contained in a series of "Contributions to the Chemistry of the Natural Hydro-carbons and their Derivatives," from the laboratory of the Barber Asphalt Paving Company, and have been re-printed, with corrections and additions, from an article in the *Journal* of the Society of Chemical Industry for January, 1898.

Form of the Lake Deposit

All that was known in 1891 of the Pitch Lake deposit was purely superficial, and showed merely that it covered an area of about 114 acres ; that the surface was in constant motion, as was proved by the appearance on its surface of stumps of trees brought up from below, which, after being carried to a certain height, would topple over, to be again engulfed and disappear ; that there were several groups of trees or islands sustained on the pitch ; that there was a pool of soft pitch of ordinary temperature at the centre where gas was evolved ; and that wherever a hole was dug in the pitch, whether deep or shallow, it filled up, and the surface resumed its level after a short time. Nothing was known as to the size or depth of the deposit, the shape of the enclosing walls, or of the amount of new material which flowed into the lake each year.

In 1893 and 1894, however, when the Trinidad Asphalt Company began to establish a pier and cableway for conveying the crude pitch from the lake to vessels for shipment, a series of borings was made upon the lake by Mr. P. W. Henry, now general manager of the Barber Asphalt Paving Company, as well as on the land between the lake and the gulf, and in the gulf to the north and west of the lake; and a line of levels was run from the shore to the lake, and across it through the centre in several directions, with stations at intervals of 100ft., all secured by bench marks on firm ground.

The boring at the centre of the lake was carried to a depth of 135ft., the entire distance being through pitch, which, as far as ocular evidence goes, has the same character as that at the surface. It was impossible to carry the boring deeper, as the movement of the pitch had so inclined the tube—1ft. in 6ft.—which formed the lining, that it had to be abandoned. It then gradually toppled over and was engulfed. Nothing has been seen of it since. The result was sufficient, however, to show the great depth of the crater and the uniformity of the pitch. The depth attained was within a few feet—not more than three and a half—of sea level, and yet we do not know how much deeper the pitch may extend. The borings on the north side of the lake, about 100ft. from the centre, and 100ft. from the edge, was in pitch of the usual character for 75ft., showing a very steep slope to the sides of the crater. At 80ft. a layer of fine white sand was met for a few feet, and then asphalt was again encountered. At 90ft. sand mixed with asphalt was struck, and this continued to a depth of 150ft.

Further borings, made at some distance from the lake, gave results near the surface which were similar to those

found at the deeper levels at the edge of the lake. Sand, mixed with asphalt here and there, was the common material, while at a depth of 80ft. on the southern side of the lake, and about 80ft. south of the road, and between 1200ft. and 1300ft. from the centre of the lake, a very hard asphaltic sandstone was found.

All the evidence thus goes to show that the sides of the crater are of sand or sandstone, more or less impregnated with bitumen, the sandstone being no doubt the rock of the hillside toward the south, against which the crater has been built up.

From the borings it was thus learned for the first time how enormous the deposit was, and the idea that the mound was really a crater seemed to be confirmed. It is, nevertheless, hard to realise that there is at this point, 138ft. above the sea, a bowl-like depression over 2300ft. across, and over 135ft. deep, reaching below the sea level, and filled with a uniform mass of pitch, which must amount to over 9,000,000 tons. Nothing less remarkable are the conclusions reached from the results of the lines of levels run across the lake.

In February, 1893, four lines of levels crossing at the centre were run across the lake and secured by plugs on the shore. Bench marks of concrete were also put in on hard clay some distance back from the lake, which in 1894 were found not to have changed their level. The results of these levels of 1893 showed that the centre of the lake was about a foot higher than that portion a thousand feet outward toward the edge, and that from the latter point to the edge was a rise of 6in. The elevation of the centre above sea level was 138'5ft.; of the station, 1000ft. N., 27 deg. W., 137'5ft.; of the edge, 1100ft [from the centre, 138'0ft.; while the top of the crater wall itself, 100ft. further on, was

140·1ft., and at the diametrically opposite side of the lake, 141·4ft. There is considerable irregularity in the height of the crater wall, due, no doubt, as suggested by Peckham, to its breaking down in part under the pressure of the pitch. The highest part, as shown in my 1892 report, is to the south, with an elevation of 141·4ft. above sea level, and the lowest toward the west and toward the north-east, where it is not less than 137·8ft. or 138ft. above sea level, the highest level probably being that of the original rim, as confirmed later.

The surface of the lake, it is very evident from even a casual examination, is lower to-day than some years ago, and the deposit now seems to occupy a shallow depression. This fall is due to the removal of the crude pitch for shipment, which has reached over a million tons. From the difference in level of the surface of the lake between any two years, and from the amount of pitch removed, it is easy to calculate how many tons of it correspond to a fall of an inch or a foot. In a similar way, from the area of the surface and the density of the pitch, it is possible to calculate on another basis how much pitch should correspond to an inch or a foot in depth. From the latter figures it appears that the lake should have fallen many more feet than it has in the last thirty years, and from a comparison of the results of the first and second calculations, extending over three periods and three sets of levels, there is no doubt that there must be an influx of fresh pitch at the soft spot, which, in the periods between 1893 to 1896, amounted to an average of from 20,000 to 18,000 tons per year. There is, therefore, no doubt that there is a vast influx of pitch into the lake at the present time, amounting to at least 18,000 long tons per year, and adding to a supply which must, from the area and depth of the

crater, reach 9,000,000 tons. The enormous size and activity of this deposit are equally striking.

Movement of the Lake Surface.

The constant movement of the surface of the lake, as shown by the course which sticks and logs take, which rise in the pitch, was well brought out in the work of running the levels which have been mentioned. Stakes driven for stations in levelling in a right line across the lake were, near the more actively moving central portion, much out of line in twenty-four hours, and within three weeks those at intervals of 100ft. for 600ft. from the centre had moved as follows:—

Centre 0	..	20·6ft. to right	..	12·7ft. ahead on line		
„ 1		13·5	„	1·1	„	„
„ 2		6·5	„	11·0	„	„
„ 3		1·7	„	0·0	„	„
„ 4		0·2	„	2·4	„	„
„ 5		0·9	„	3·8	„	„
„ 6		3·2	„	1·4	„	„

On one of the lines there was an island or mass of floating vegetation which was marked by a hub in 1893. In 1894 this island, 600ft. from the centre of the lake, had moved 5·5ft. to the left of the line, and 23ft. in the direction of the line toward the edge of the lake. The positions of the islands intersecting the lines of levels were also determined in 1896, and all of them found to have shifted their position.

By these determinations, therefore, the activity of the surface movement of the lake is definitely settled.

Proximate Composition of Trinidad Asphalt.

When in Trinidad, in 1891, Mr. Richardson made a collection of specimens of the crude pitch, or asphalt, at

intervals of 200ft., on lines laid out on two diameters of the lake. These were examined according to the methods in use at that time, the water having been previously removed by drying at 100 deg. Cent., and the solvents being applied at ordinary temperatures. The results obtained showed great uniformity in the composition of the crude asphalt, but were not entirely satisfactory for several reasons. The specimens were taken too near the surface of the lake; they were liable to have suffered an alteration and loss of light oils by being dried at too high a temperature; and the more recent methods of analysis, with the use of light naphtha as a solvent, had not been elaborated. He found soon after that the emulsified water in the pitch could be removed very readily, without the aid of heat, by grinding the pitch to a fine powder and exposing it to the air. The rapidity with which the water evaporates and the ease with which the pitch dries is illustrated by the following determinations. A piece of crude pitch was weighed, ground, and passed through an 80-mesh sieve, and then exposed in a thin layer to the air of the laboratory. The loss of water was as follows:—

Loss in	Per cent	Per cent. of entire water.
5 minutes	2·2	7·0
12 ,,	3·5	12·1
20 ,,	6·5	22·4
30 ,,	9·5	32·7
1 hour	16·5	56·9
2 hours	20·0	69·0
3 ,,	21·0	72·4
and after regrinding		
4 hours	22·5	77·6
24 ,,	29·0	100·0
and in vacuo over H ₂ SO ₄	0·6	—

On exposure to air saturated with moisture, the powder of

crude pitch, after final drying in vacuo, gained in twelve hours 2·8 per cent., an amount of hygroscopic moisture which is not large, and which is quite different from the emulsified water originally present, which evaporates as soon as the bituminous cells which enclose it are broken down. In fact, the manner in which the water exists in Trinidad asphalt is quite different from that in which it is found in most other substances occurring in Nature, since it cannot be removed by diffusion or osmosis, as in the case of drying a lump of clay or a vegetable structure, but only by breaking down the enclosing cell wall of bitumen. The novelty of this method of drying the crude asphalt was acknowledged by the Patent-office, and a patent granted therefor. The peculiarity has since been noticed by other investigators. Owing to the ease with which the water can be removed from Trinidad Lake asphalt in this way, a means was afforded of collecting the pitch and drying it at the lake, so that no change could take place in transit.

A final series of samples was collected in this way by Mr. P. W. Henry during his work at the lake in February, 1894. In order also to obtain true representative samples of the material forming the mass of the deposit, the surface samples were taken about 2ft. below it, and others from the borings which have been mentioned as extending as deep as 135ft. at the centre and 150ft. on the edge. Mr. Richardson was thus supplied with a set of samples which fairly represent the true character of the crude pitch as it exists in the lake. These samples were examined as follows:—

Method of Analysis.

Separate weighed portions of the material dried in vacuo over sulphuric acid were extracted in beakers with successive portions of hot carbon bisulphide and naphtha of 88 deg. B.P.,

the decanted solvents being passed through a Gooch crucible with heavy asbestos felt. The filtrate was allowed to settle, in the case of the carbon bisulphide extract for twenty-four hours, again decanted, and any fine sediment which had passed the filter brought upon it. The losses represented the bitumen soluble in the two solvents. The mineral matter was determined by direct ignition, and the organic matter not soluble by difference. This determination by difference is too large, owing to too great loss on ignition in the determination of the mineral matter; and too small, owing to the calculation as bitumen of some substance that is left in the bisulphide solution which is not bitumen. The two errors nearly neutralise each other, although in more recent analyses the carbon bisulphide solution is finally burned and ignited, and the amount of mineral matter found subtracted from the loss taken as the amount of bitumen.

Great care is essential in these determinations, especially that the solvents be perfectly dry. On this account bisulphide is much more suitable for use than oil of turpentine or chloroform, as it is not nearly so hygroscopic, although it is not quite as complete a solvent unless used hot.

Examined in this way, the results given on page 182 were obtained for the 1893 collection.

In these analyses of the surface samples an even more striking uniformity in composition is found than in those collected in 1891, owing to the care in their collection and preparation, and to the method of analysis. The amount of bitumen is somewhat higher, on account of the more thorough extraction (to day, with still further improvements in the mode of treatment, it would be even more so), but relatively Mr. Richardson found the same evidence of a

fixed proportion of bitumen, mineral matter and organic matter not bituminous.

In the samples from the boring at the centre of the lake, which extended to a depth of 135ft., and was still in asphalt, there is not as great uniformity, because of the way in which it was necessary to collect them by washing the particles detached by the boring machine up through

Average Composition of Trinidad Lake Pitch in Circles.

From centre.	Bitumen by CS ₂ .	Mineral matter.	Organic, not soluble.	Soluble in Naphtha.	Percentage of total bitumen thus soluble.
	Per cent.	Per cent.	Per cent.	Per cent.	
Circle 2, 200ft.	55·02 ..	35·41 ..	9·57 ..	31·83 ..	57·85
Circle 4, 400ft.	54·99 ..	35·40 ..	9·61 ..	31·63 ..	57·55
Circle 6, 600ft.	54·84 ..	35·49 ..	9·67 ..	31·85 ..	58·26
Circle 8, 800ft.	54·66 ..	35·56 ..	9·78 ..	31·67 ..	57·97
Circle 10, 1000ft.	54·78 ..	35·44 ..	9·78 ..	31·58 ..	57·64
Circle 12, 1100ft.	54·62 ..	35·45 ..	9·93 ..	31·77 ..	57·51
General average	54·92 ..	35·46 ..	9·72 ..	31·72 ..	57·79
Circle 14, 1400ft.	53·86 ..	36·38 ..	9·76 ..	30·52 ..	55·66

Average Composition of Trinidad Lake Pitch from the Boring.

..... 54·66 .. 35·90 .. 9·44 .. 31·53 .. 57·67

the bore with a current of water, and catching the material in a pail, where it was allowed to settle and the sample taken. Nevertheless the results are sufficiently close to show that the material at all depths is the same, and when they are averaged the agreement between the composition at the surface and for an average depth is remarkable.

The average composition of the two lots of sample from the surface and from the boring is as follows:—

	Bitumen.	Mineral.	Organic.	Bitumen soluble in naphtha.	Total bitumen. Per cent.
Surface ..	54.92	.. 35.46	.. 9.72	.. 31.72	.. 57.79
Boring ..	54.66	.. 35.90	.. 9.44	.. 31.53	.. 57.67

At the side of the lake the boring, about 100ft. from the edge of the crater, showed similar pitch to that on the surface for 75ft., after which there was, as has been mentioned, a change, and the composition of the borings proved to be that of sand and soil mixed with asphalt, showing that the side of the crater had been reached.

The uniformity of the proximate composition of all the pitch that exists in the lake and is newly forming there leads to the belief also that the name Parianite, suggested for it by Prof. Peckham, as a mineral species, is entirely justified.

Bitumen Soluble in Naphtha.

In Mr. Richardson's report of 1892 he showed that between the refined products of that pitch from the Trinidad Lake and that from deposits outside of the lake, known as "land asphalt," a decided difference could be detected in the relative amount of their total bitumen soluble in 88 deg. naphtha.

Determinations, in the same way, of the amount soluble in the lake samples, have proved of interest in connection with the comparison of those from the surface and from the borings. The average percentage of bitumen soluble in naphtha, in both the surface samples and those from the boring at all depths, is the same, or nearly so—57.79 and

57·67 per cent. ; but on the surface itself the amount is found to diminish somewhat toward the edge of the lake and in the boring the average for the first 70ft. is 56·83 per cent., while for the second it is 53·50 per cent. The effect of age is seen in these cases much in the same way as, to a much greater extent, is found to be the case with the land asphalt.

The Deposits of Land Asphalt.

Beyond the boundaries of the Trinidad pitch lake, and between it and the sea, and even in the sea itself, are found deposits of crude pitch very similar to that in the lake, or in some stage of alteration which permits of their recognition as being originally derived from such material.

There is no doubt that a large area to the north and east of the lake contains large quantities, and it has been an open question as to how it got there. Manross describes an overflow from the lake, and to this is undoubtedly due the presence of pitch on the lands adjoining it and for some distance toward the sea to the east, but a large amount of the pitch must have been ejected independently of the lake source of supply, but so long ago that it has been buried with soil for years. There has been no overflow for years, nor any evolution of fresh pitch at any point in the neighbourhood of the lake, except one or two small cones, so that there is no activity to-day which will reveal what happened in past centuries. The pitch has reached the seashore in many instances, and even forms reefs beyond it. It is spread out on the Point d'Or estate to the east of the lake, evidently in a large sheet, which seems more like an evolution of pitch independent of the lake

supply than any other. This has been overgrown for some time, and its age, which can only be determined by the changes which analysis would show it to have undergone, is doubtful.

Where the pitch comes in contact with salt water it is hardened and does not rot, and to this cause are due the asphalt pebbles of all sizes and shapes found upon the beach. Where exposed to the alternate action of the sea water and air the pitch is converted into alteration products, which will be noticed later.

It is evident that the pitch forming the land deposits is, as a whole, of a very varied character, and to be found in all stages of alteration.

Proximate Composition and Properties of Trinidad Asphalt from the Land Deposits.

From the results of the examinations of the specimens of pitch collected in 1891 from the land deposits, the conclusion was drawn that the relative proportions of bitumen, organic matter not soluble, and inorganic or mineral matter, in this form of pitch were not far different, in carefully selected material from which all oxidised and altered pitch had been excluded, from those found in the lake substance. The amount of water, however, proved to vary somewhat in the land pitch, and in the dry material the proportion of the entire bitumen which was soluble in naphtha of low specific gravity—0.63 to 0.64—was shown to be decidedly smaller in the land than in the lake samples, while the physical properties of the two kinds of asphalt also proved different.

Examinations of a collection of representative land pitch specimens resulted as follows.

*Average Composition of Lake Pitch, Dried in Vacuo,
Kearney Collection.*

	Bitumen- soluble CS ₂ .	Mineral matter.	Organic not soluble.	Bitumen- soluble petroleum naphtha.	Per cent. of total Bitumen- soluble in naphtha.
	Per cent.	Per cent.	Per cent.	Per cent.	
Average ..	54·25	36·51	9·24	35·41	65·27

Eight Specimens from Lot C., near the Lake.

Average ..	54·03	36·49	9·48	33·02	61·11
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Four Specimens from Crown Land Lots Adjoining C.

Average ..	53·81	36·62	9·57	32·29	60·01
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Five Specimens from East of Road, Middle Ground.

Average ..	52·31	37·80	9·89	31·25	59·74
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Seven Specimens from Village Lots, near the Gulf.

Average ..	52·27	37·73	10·01	31·42	60·12
General average ..	53·10	37·16	9·74	31·99	60·14

These averages enable us to compare the composition of the pitch from various parts of the deposits outside the lake among themselves, as well as with that of the lake pitch analysed under similar conditions. It appears in the most striking way that the further from the lake the pitch is found, the more it shows signs of age, as evidenced by the increase of the percentage of organic matter not soluble, that is to say, of altered bitumen and of mineral matter, and, generally, a decrease in the per cent. of the total bitumen, which is soluble in the naphtha used. The lake pitch has 65·3 per cent. of its bitumen soluble in naphtha, while just outside the lake the land pitch has only 61·1, and further on only 59·7 per cent. soluble. This

may seem a small difference, but it is evidence of a large change. In glance pitch, examined in the same way, 49 per cent. only of the entire bitumen was found to be soluble in naphtha, in lake pitch 65·3. Land pitch may, therefore, be inferred to be about a quarter converted from lake to glance pitch.

The relation between the composition of the two kinds of pitch has also been shown in comparing the results of analyses of some land samples. The three sets of material were found to have the following average composition:—

Comparative Composition of 1894 Samples.

	Bitumen.	Organic insoluble.	Mineral inorganic.
Lake, 2ft. below surface ..	54·92	9·72	35·46
Lake boring, 135ft. deep at centre	54·66	9·44	35·90
Land	52·36	11·24	36·40
	Bitumen soluble in naphtha.	Total bitumen soluble in naphtha. Per cent.	
Lake, 2ft. below surface.. .. .	31·72	..	57·79
Lake boring, 135ft. deep at centre	31·53	..	57·67
Land	29·02	..	55·43

The same relative differences are seen as in the previously-mentioned series.

Again, we have the evidence of another investigator, Professor S. F. Peckham, who gives, in the *American Journal of Science* for March, 1896, a series of analyses of specimens of crude lake and land pitch, which he collected personally in 1895. Mr. Richardson has re-arranged these analyses, classifying them according to their lake or land origin, and leaving out one analysis of lake pitch, No. 21, which is plainly in error. Averages derived from these

analyses, which are as follows, although carried out on somewhat different lines, permit of the same conclusions in regard to the changes which have taken place in the land samples:—

Crude Lake Pitch.

Percentage soluble in				Percentage of total bitumen only soluble in			
Petro- leum ether.	Boiling spirits turpen- tine.	Chloro- form.	Total bitu- men.	Petro- leum ether.	Boiling spirits turpen- tine.	Chloro- form.	
Average ..	35·2 ..	12·4 ..	5·2 ..	52·8 ..	66·8 ..	23·3 ..	9·9

Crude Land Pitch.

Average ..	33·3 ..	11·9 ..	6·5 ..	51·7 ..	64·7 ..	23·0 ..	12·3
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Iron Pitch.

No. 3 ..	33·6 ..	13·8 ..	9·9 ..	57·2 ..	58·7 ..	24·1 ..	17·2
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The determinations of bitumen made by Professor Peckham with chloroform also furnish some additional and conclusive evidence of the differences between the two kinds of material. These show that the average land specimen contains 2·4 per cent. more of its bitumen in this difficultly soluble form, while iron pitch, which is acknowledged to be of no value for paving purposes, and is always rejected in digging land pitch, has 7·3 per cent. more of its bitumen in this form. From this Mr. Richardson draws the inference that the land pitch collected by Professor Peckham is one-third converted into iron pitch, and of so much less value than lake pitch for paving purposes. In fact, his results are as conclusive evidence of the differences between lake and land pitch as any that have been offered, and confirm the results of experience with the asphalt from the land deposits in the laying of street pavements.

Composition of the Soft Pitch and Fitch from Blow Holes.

As has been shown, the soft pitch, at the centre of the lake, is in an active state of change, so that it might readily prove to be somewhat different in composition from the general run of pitch from the lake. The same thing might be the case with the asphalt found in a few active blow-holes at or near the edge of the deposit.

Specimens of the soft asphalt have been examined, with the following results—the 1891 material being analysed a few weeks after it was collected and while still in an active state of change; the 1893 specimens after they had ceased to give off gas, and were still of rather soft consistency, and easily cut with a knife:—

	1891.	1893.	1893.
Water at 100 deg. Cent.	—	(28.9)	—
Loss on melting	34.1	31.4	—
Bitumen in the fresh pitch	34.5	43.6	—
Organic matter insoluble in CS ₂	6.4	1.9	—
Mineral matter	25.0	23.2	—
	<u>100.0</u>	<u>100.0</u>	<u>—</u>
On dry substance :			
Bitumen	52.4	63.4	60.2
Organic insoluble	9.6	2.8	6.9
Mineral matter	38.0	33.8	32.9
	<u>100.0</u>	<u>100.0</u>	<u>100.0</u>

The three specimens differ considerably. In the first, the composition is similar in the dried condition to that of average lake sample, having, however, more mineral matter. In the two others there is less mineral and organic matter not bitumen, with a consequent increase in bitumen. It would seem, therefore, that the soft pitch is not as uniform in composition at the source of the lake's supply as that of the main mass of pitch, but by the continued movement of

the mass becomes later of the average found in other parts of the deposit.

There is also, in the case of the soft pitch, a loss of gas and light oil on heating and melting which does not take place with the hard asphalt to any such degree, and on allowing the soft and hard pitches to flow at their softening points on an inclined plane of brass, the former moves 5·8in. where the hard runs but 3·8, or 53 per cent. further. This happens even after the soft pitch has been several years out of the lake and becomes decidedly hardened both by age and melting to drive off water.

Samples of the comparatively soft pitch from a blow-hole near the power station at the edge of the lake and but a few feet back from the main mass were examined to determine if it was from the same source, and had the same composition as the lake asphalt. Following are the results :—

Dried material.	Per cent.
Bitumen	54·0
Organic, not soluble	9·1
Mineral matter	36·9
	100·0

The blow-holes, of which there are two or three near the lake, must be connected with the source of the main supply, but they evolve a pitch unaccompanied with free water and different from that welling up at the centre of the lake, being more like the average lake pitch.

Water in the Pitch and in the Lake.

The analyses of the crude pitch in 1891 showed that there was a loss, on heating the samples that had been brought from the lake, without drying, to 100 deg. Cent., of from 25·8 to 30·6 per cent. This was, of course, largely water, but in some cases, at the comparatively high temperature

employed, some gas and light oil was driven off, while in others there had been a loss of water in transit. In the dried samples of 1894 it was, of course, impossible to determine the water, but this has been done with carefully taken samples of the crude pitch from large and fresh cargoes as soon as they were discharged in New York. In this way it has been found that the amount varies but little from the limits, 28·5 to 29 per cent. On this basis the composition of the crude pitch, although analysed in a dried condition, was :—

Water	28·5
Mineral matter.. .. .	25·4
Organic, not soluble in hot carbon bisulphide	6·9
Bitumen	39·1

Of the character of the water in the crude pitch, it was shown in 1892 that it was strongly mineral. Attempts to collect it in any other way than by melting the crude pitch and allowing it to rise to the surface have been unsuccessful, although it would be more satisfactory to collect it at ordinary temperatures and without condensation or change. As thus secured from the still in which the fire-refined asphalt is produced, and in a somewhat concentrated condition, it had the following characteristics :—

	Grms. per litre.
On evaporation the residue was equivalent to ..	19·872
Dried at 100 deg. Cent.	17·484
„ 130 deg. Cent.	17·006
Ignited gently	15·014

These specimens of water from the stills must, of course, have become somewhat concentrated beyond their normal condition, but they illustrate its nature as well. It contains, as most striking constituents, iodides, and borates (characteristic of thermal water), and ferrous sulphate, which gives it a

strong acid reaction. There is also a remarkably large percentage of ammonia, the presence of which is of great interest. The principal salts are chlorides and sulphates, and its acidity is sufficiently great to attack and corrode deeply the steel buckets in which the crude pitch is brought from the lake to the shore. In fact, the ground and surface water, wherever it comes in contact with the crude pitch, acquires an acid reaction, and has been found unsuited for use in boilers.

Mineral Matter in the Pitch.

The mineral matter in Trinidad asphalt consists of silica principally, accompanied by some clay, oxide of iron, and the substances soluble in the water of the crude pitch.

An analysis of the entire ash left on burning the crude pitch resulted as follows :—

	Soluble in acid.	Insoluble.	Total.
Silica, SiO ₂	—	70·64	70·64
Alumina, Al ₂ O ₃	7·38	9·66	17·04
Ferric oxide, Fe ₂ O ₃ *	6·30	1·32	7·62
Lime, CaO	0·46	0·24	0·70
Magnesia, MgO	0·11	0·79	0·90
Soda, Na ₂ O	1·56	—	1·56
Potassium, K.	0·35	—	0·35
Sulphuric oxide, SO ₃	0·97	—	0·97
Chlorine, Cl.	0·22	—	0·22
	17·35	82·65	100·00

* FeO not determined.

The silica under the microscope appears in the form of flakes with sharp fracture. It seems probable, as suggested by Dr. Carl Barus and Professor Peckham, that it must have been deposited from solution in the water found in the pitch. The particles are naturally much larger than those of the rest of the mineral matter, although in themselves

very small. By treatment of the ash with strong acid to remove the soluble portion, the silica can be obtained in a white condition, but mixed with some impalpably fine white clay, which can be separated by decantation. It will be seen that the insoluble portion of the mineral matter of Trinidad pitch contains 85.4 per cent. silica.

Some of the mineral matter is so impalpably fine that it will not separate from a solution of melted or dried Trinidad pitch in any of the usual solvents even after days of standing and many hours' treatment in a centrifugal. It will pass also through the finest filters. It has been thought by Peckham and others, on this account, to be chemically combined with the organic compounds of the asphalt, but Mr. Richardson found that by continued swinging in a centrifugal it can be so far reduced that it amounts to but 2 per cent., and is then, apparently, only in a state of mixture with the bitumen, since an analysis of the very finest portion recovered by burning the pure bitumen thus swung out shows that it is a ferruginous clay, and could not possibly be combined with organic matter, since it consists of silicate of alumina, and a very considerable amount of sulphuric acid, as well as oxide of iron.

Analysis of Finest Mineral Matter.

	Insoluble in HCl.	Soluble.	Total.
SiO ₂	32.36	32.36
Al ₂ O ₃	6.74	33.64	40.38
Fe ₂ O ₃	1.40	11.74	13.14
CaO	0.45	3.20	3.65
MgO	0.34	1.40	1.83
K ₂ O	—	1.18	1.18
Na ₂ O	—	0.53	0.53
O ₃	—	7.16	7.16
	<hr/>	<hr/>	<hr/>
	41.29	58.94	100.23
			0

The Bitumen of Trinidad Asphalt.

The bitumen of Trinidad asphalt can be separated from the mineral matter and organic matter not bituminous by solvents, and, as has been seen, amounts to about 39 to 40 per cent. of the crude pitch, or 54 to 57 per cent. of the dried substance. For purposes of investigation, it is more safely extracted with chloroform at a boiling temperature. It is freed from suspended mineral matter by subsidation and long treatment in a centrifugal machine, and from traces of the solvent by heating to 200 deg. C. for a short time with stirring. As thus prepared, it contains about 2·3 per cent. of clay and iron oxide, which, thus far, it has been impossible to remove.

The total bitumen of Trinidad asphalt is a brilliant, glossy, pitch-like substance, which has a semi-conchoidal fracture when struck a sharp blow, but which yields to gentle pressure and slowly flows at summer temperatures. It softens rapidly at 76 deg. C., and flows quickly at 83 deg. C., but is not liquid until above 100 deg. C. It has a specific gravity, as extracted, of 1·071 at 25 deg., and, after correction for the 2·6 per cent. of mineral matter present, of 1·032.

The ultimate composition of several preparations which involved complete extraction was determined with the precautions necessary in burning such organic substances, and gave the following results:—

Total Bitumen in Trinidad Lake Asphalt.

Preparation.	I.	IV.	V.	Average.
Carbon	82·59	81·95	82·44	82·33
Hydrogen	10·74	10·51	10·81	10·69
Sulphur	6·04	6·54	5·90	6·16
Nitrogen	0·51	0·92	1·00	0·81
	<hr/>	<hr/>	<hr/>	<hr/>
	99·88	99·92	100·15	99·99

This bitumen is characterised by the large percentage of sulphur which it contains, and the presence of nitrogen. There are apparently no oxygen derivatives present in the bitumen, or they occur in very minute amounts.

The Bermudez Asphalt Deposit.

From the mouth of the Orinoco, the north-eastern coast of Venezuela, which faces Trinidad, is low, and consists of vast mangrove swamps, through which run deep tidal estuaries. That portion forming part of the State of Bermudez extends inland for many miles. It lies on the opposite side of the Gulf of Paria from Trinidad. About thirty miles in an air line from the coast the asphalt deposit, known as the Bermudez Pitch Lake, is found at the point where a northern range of foot hills comes down to the swamp. The Guanaco River, a branch of the San Juan, one of the large caños or estuaries of this region, at about sixty-five miles, in its winding course, from its mouth, runs within three miles of the deposit, but it is five or six miles to a suitable wharfage site. On the other hand, towards the north a road runs to the hills and to the village of Guaryquen. These are the means of communication with the deposit. The so-called lake is situated between the edge of the swamp and the foot hills in what might be termed a savanna. It is an irregular-shaped surface, with a width of about a mile and a-half from north to south and about a mile east and west. Its area is a little more than 900 acres, and it is covered with vegetation, high rank grass and shrubs, 1ft. to 8ft. high, with groves of large moriche palms, called morichales. One sees no dark expanse of pitch on approaching it as at the Trinidad pitch lake, and except at certain points where soft pitch is welling up, nothing of the kind can be found. The

level of the surface of the deposit does not vary more than 2ft., and is largely the same as that of the surrounding swamps. In the rainy season it is mostly flooded, and at all times very wet, so that any excavation will fill up with water. These conditions make it difficult to get about upon it or to excavate pitch easily.

It is readily seen that this deposit is a very different one from that in the pitch lake of Trinidad. It seems to be, in fact, merely an overflow of soft pitch from several springs over this large expanse of savanna, and one which has not the depth or uniformity of that at Trinidad.

Being on a level with the mangrove swamps, and with foot hills on its other side, any large amount of asphalt could hardly be held in position here, as in the old crater in Trinidad, but would burst out into the swamp and be lost, and, as far as borings have been made, they seem to indicate but a small depth anywhere as compared with that of the Trinidad lake.

At different points there is at most a depth of 7ft. of material, while the deepest part of the soft maltha is only 9ft., and the average of pitch below the soil and coke only 4ft. At points there is not more than 2ft. of pitch, and in the morichales or palm groves it is often 5ft. below the surface. At several points scattered over the surface are areas of soft pitch, or pitch that is just exuding from springs. The largest area is about 7 acres in extent and of irregular shape. This has little or no vegetation upon it, and, from the constant evolution of fresh pitch, is raised several feet above the level of the rest of the deposit. This soft asphalt has become hardened at the edges, but when exposed to the sun is too soft to walk upon. The material is of the nature of a maltha, and is evidently the source of all the asphalt in the lake, from these exudations the pitch having spread

in every direction, so that no great depth of pitch is found even at this point.

A careful examination of the surroundings shows that in one respect there is a resemblance between the point of evolution of the soft pitch at the Bermudez and at the Trinidad lakes. Gas is given off in considerable quantities at both places, and in both cases consists partly, at least, of hydrogen sulphide. At the Bermudez lake Mr. Richardson was unable to determine whether it was accompanied by carbonic dioxide, but the odour of hydrogen sulphide was strong.

The consistency of the soft pitch at the centre of the Bermudez lake is much thinner than that of the Trinidad lake. It will run like a heavy tar, and does not evolve gas in the same rapid way or harden as quickly after collection. It therefore does not retain the gas which is generated in it, nor does the deposit as a whole do so to the same extent as the Trinidad pitch. Where, however, the surface of the soft pitch has toughened by exposure to the sun and air, and where gas is given off beneath it, it is often raised in dome-like protuberances, the beehives which were spoken of by visitors to the Trinidad lake. These have a thin wall of pitch, and are filled with gas which readily burns, and have been seen 2ft. or more in height and 18in. in diameter. They are, of course, found only near the soft spots.

Although the pitch at the Bermudez lake is too soft to entangle and hold permanently the gas which is given off, where the pitch of medium consistency is covered with water it does not escape so readily, and thus often raises in the pools of water a mushroom-like growth of pitch by the reduction of the gravity of the mass from the included gases. These mushrooms correspond completely, except in size, with those described by Manross as existing at the

Trinidad lake when he visited it. It seems, therefore, that we have to-day several of the phenomena represented at the Venezuelan lake which the hand of man has destroyed at Trinidad.

There is, however, no evidence of the same simultaneous boiling up of water with the fresh soft pitch that has been determined at the Trinidad lake, but that there is none at all is not certain, as at the time Mr. Richardson visited the locality heavy rains were falling which prevented the detection of a small amount. It seems, however, improbable, as the soft pitch contains little or no water, and the traces found in the samples collected are probably derived from rain.

Hardening of the main mass of pitch.

The soft pitch after it exudes at the centre of the Bermudez lake undoubtedly hardens slowly on exposure, but the condition of the surface of the main mass, which is very hard and rough, and of the harder borders of the soft spots, is due to other causes also.

The edges of the areas of soft asphalt are covered here and there with masses of glance pitch and with black and brittle cinders or coke, and which seem to have been produced from the maltha by fire. This is evidently the case, since the rank growth of grass, which is very dry in the dry season, is particularly adapted for a rapid and intense combustion. Such fires have been even recently started intentionally and accidentally, and to them are due the condition of the present surface of the deposit and the character of much of the pitch.

The general surface of the lake is very irregular and hard. There are many very narrow and irregular channels or depressions from a few inches to 4ft. deep, filled with water,

and not being easily distinguished, one often falls into them. At the foot of the growth of grass and shrubs are ridges of pitch mingled with soil and decayed vegetation, which have been plainly coked and hardened by fires of the nature which have been mentioned. When this hardened material which forms only a crust is removed, asphalt of a kind suitable for paving is found. The crust is from a foot and a-half to two feet in depth, and very firm, while the asphalt underneath would not begin to sustain the weight which that of the Trinidad pitch lake does easily. There are breaks in the crust here and there through which soft pitch exudes, as has been described.

It appears, therefore, that the Bermudez deposit owes its existence to the exudation of a large quantity of soft maltha, which is still going on, and which has spread over a great extent; that this has hardened spontaneously in the sun and has also, by the action of fire, been converted over almost the entire surface into a cokey crust of some depth, beneath which the best material lies, and that here and there are scattered masses of glance pitch produced in a similar way from less violent action of heat. There is no evidence of a general movement and mingling of the mass of this deposit in any way that would produce a uniformity of composition as seen in the Trinidad pitch lake, although there is a certain amount of gas evolved at the soft spots where maltha exudes and some gas cavities are found in the general mass of the pitch beneath the crust.

As the asphalt below the crust of the deposit is the only portion of value for paving, the question has arisen as to its uniformity. In order to determine this a collection of samples was made at various points on the lake beneath the crust, and they have been submitted to analysis.

Extremes of Determinations in the Analyses of Bermudez Asphalt.

CRUDE MATERIAL.

	Highest.	Lowest.
Loss four days, 212 deg. Fah.	46·20	10·72
Loss seven hours, 400 deg. Fah.	13·60	4·72

DRIED MATERIAL.

Loss at 400 deg. Fah., seven hours	16·05	5·81
Softens, deg. Fah.	170 deg.	140 deg.
Flows, deg. Fah.	188 deg.	135 deg.
Mineral matter	3·65	0·50
Organic not soluble	6·45	0·62
Bitumen	98·52	90·65
Bitumen soluble naphtha	73·05	63·40
Per cent. of total thus soluble	76·55	67·78
Specific gravity	1·075	1·005

Methods of Analyses.

Necessity has compelled the adoption of somewhat different methods of analyses with the Bermudez samples from those employed in the case of the Trinidad material, since the sticky nature of this asphalt prevents its being freed from water by powdering and exposure to the air. It was only possible, therefore, to dry the samples by heating them to such a point as to soften the bitumen and remove the water, this point being kept as low as possible, but undoubtedly with the loss of gas and hydrocarbons. The dried samples were then used for analysis. In addition, the amount of water, gas, and volatile oil lost by the crude material on maintaining it for a certain length of time at 212 deg. and 400 deg. Fah. was determined, as well as the loss suffered by the dried material at the latter temperature. The softening and flowing point and the degree of softness at ordinary temperatures were also determined, 1 softest, 2 soft, 3 medium, and 4 hard, as

well as the specific gravity of a few samples. The determinations of the loss at high temperatures are not as accurate as the others, but with such wide variations as were found, is of interest and value.

From the preceding results of analyses we see that the Bermudez asphalt is quite a different material from that of the Trinidad lake. It is practically free from mineral matter: in fact, the softest material from the soft spot at the centre, before it was in any way contaminated with extraneous matter, has no more than 0.02 per cent. It has, as collected, varying amounts of water, and the loss on heating to 100 deg. C. for some time may reach as high as 46 per cent. Here again, however, it appears from an examination of the freshly collected softest material that the water is adventitious and the loss largely light oils, as this soft material contains but a trace of water. The amount of organic matter insoluble is, probably, also adventitious, as this is not found in the soft pitch, but it may in part be derived from changes in the bitumen itself from age and exposure. The portion of the total bitumen soluble in naphtha is large, but not constant in the specimens from the different parts of the lake, but of the fresh material nearly the entire amount is soluble.

A mere glance at the results then shows that there is a great lack of uniformity in the condition and character of the material found scattered over the surface of the Bermudez deposit, and it appears that this bed of asphalt is one that was originally, and is now being, derived from exudations from maltha springs, scattered over the area which the deposit covers; that the original thick oil or maltha was a pure bitumen free from mineral matter and water which has become altered by changes within itself and by external causes, until to-day it exists in the form

found in the so-called lake. There is here, as in the Trinidad pitch lake, a present source of supply which is adding every year to the main body of the pitch.

Character of the Bitumen of Bermudez Asphalt.

An investigation of the nature of the bitumen of Bermudez asphalt has been partially carried out in the same way as with the Trinidad material, and has furnished some results of interest.

The softest and freshest asphalt from the point where it exudes has the following composition:—

	I.	II.
Carbon	82·88	—
Hydrogen	10·79	—
Sulphur	5·87	5·37
Nitrogen	0·75	—
	<hr/>	<hr/>
	100·29	—

The striking features of this bitumen are the very high percentage of sulphur, nearly as great as that in Trinidad bitumen, and the notable percentage of nitrogen, while in other respects it shows only evidences of being composed largely of saturated and unsaturated alicyclic hydrocarbons. On comparing the original bitumen with that found in the general supply of the lake, as used for pavements, an interesting change is seen. This bitumen, extracted from the melted and refined pitch by chloroform, has the following composition as derived from the combustion of two different preparations:—

Entire Bitumen Bermudez Paving Asphalt.

	I.	II.
Carbon	—	—
Hydrogen	—	—
Sulphur	3·93	3·41
Nitrogen	0·75	0·82

Here a remarkable alteration is seen. The hardening of the pitch or, very likely, the process of melting and refining has removed a very considerable part of the sulphur, nearly 2 per cent. The sulphur must have been given off as hydrogen sulphid \bar{e} , and in its evolution condensation has taken place, and a consequent hardening of the bitumen.

The nitrogen remains unchanged.

Sulphur, therefore, in this case, as in that of Trinidad asphalt, seems to play an important part in the hardening of the bitumen. It is also found in larger amount in that portion of the Bermudez bitumen which is insoluble in naphtha but soluble in chloroform, as with Trinidad asphalt, the bitumen soluble only in chloroform having the following composition :—

	Per Cent.
Carbon	87·19
Hydrogen	8·47
Sulphur	4·83
Nitrogen	—
	<hr style="width: 10%; margin-left: auto; margin-right: 0;"/>
	100·49

Sulphur in Asphalt, and its Influence.

The interest which the presence of so much sulphur in the bitumen of Trinidad and Bermudez asphalt, and especially in the least soluble and harder portions, aroused led me to examine a large number of asphalts and malthas of varied origin, which were available in the collection of the Barber Asphalt Paving Company, with a view of determining how general the presence of a large percentage of sulphur is in bitumens, which are hard and of high melting point.

At the same time complete ultimate analyses were made of these bitumens, in order to show that oxygen is not to

any extent a constituent of that portion of an asphalt soluble in chloroform or bisulphide of carbon, to determine how generally nitrogen is one, and what differences there may be in the saturation of the hydrocarbons.

The determination of sulphur was made on the pure bitumens after the method, described by Mabery, of combustion in a current of oxygen, and absorption of the sulphur dioxide formed in a standard solution of alkali. In several instances, especially Trinidad pure bitumen, the results were checked by determinations made by oxidation with nitric acid. Mr. Richardson's experiments and those of Hodgson have shown that none of the ignition methods with carbonates, magnesia, or sodium peroxide give high enough results.*

The analyses for carbon and hydrogen were made with all the precautions which a long experience with this kind of work has shown to be necessary. They were determined by combustion with manganese oxide Mn_2O_3 with a layer of lead chromate and a copper coil on account of the presence of sulphur and nitrogen. The copper coil and the substance in a platinum boat were introduced into the tube after the tube and its contents had been raised to a red heat in current of oxygen and allowed to cool. The combustion was carried on very slowly in a stream of oxygen, and the absorption of the water and carbonic acid carefully provided for and protected. Nitrogen was determined readily by the Kjeldahl method.

The results are shown on pages 206 and 207.

It appears in a general way from the preceding results that all the glance pitches, or brittle asphalts, Nos. 1, 3, 4, and 5, contain from 9.76 to 8.28 per cent. of sulphur, the

* J. Am. Chem. Soc. 20, 882.

hard asphalts, not flowing at ordinary temperatures, from 6.47 to 3.93 per cent., while the malthas or soft asphalts and heavy petroleums do not contain more than 2.29 per cent. There are, however, certain exceptions. The soft Bermudez and Montana asphalts should, from their consistency, be classed with the malthas, but are placed by themselves, because they are rapidly transformed under certain conditions to their harder forms, which are included under the asphalts, probably because of the high percentage of sulphur they contain.

The Uvalde County bitumen is also an exception, in that it is of only medium hardness, although having 9.60 per cent. of sulphur. This bitumen is, however, peculiar, and differs from the asphalts in some of its other properties as well.

Those natural bitumens also, which are usually classed by themselves, albertite, gilsonite, grahamite, wurtzelite, and ozokerite, are distinguished by a low percentage of sulphur and a high percentage of carbon, with the exception of wurtzelite, which appears, therefore, to be more nearly allied to the asphalt. They are probably, therefore, of different origin from the asphalts, and as an illustration of such a difference, the origin and method of formation of the artificial asphalts included in the table will serve.

The so-called Pittsburg flux is produced by heating heavy petroleum oil, such as Pennsylvania residuum, with sulphur. Hydrogen sulphide is given off, and the oil is condensed to a peculiar tough and sticky substance, but slightly affected by temperature changes, and only melting at a very high temperature, which has been suggested as a substitute for asphalt. Here the formation of the artificial asphalt is due to the condensation of olefine and paraffin hydrocarbons by the action of sulphur, at a high temperature, and while much is evolved, several per cent. are left in the product.

ULTIMATE COMPOSITION OF THE BITUMEN OF ASPHALTS, MALTHAS, &c.
Hard Asphalt.

	Consistency.	In extracted bitumen.					Total.
		Sulphur.	Carbon.	Hydrogen.	Nitrogen.		
Nevada	Pitch	9'76	79'58	9'31	1'30	99'95	
Texas, Uvalde County	Hard	9'60	80'32	10'09	0'28	100'20	
Italy, Ancona	Pitch	9'39	79'56	8'80	1'40	99'15	
Egypt	"	8'52	80'87	10'42	0'19	100'00	
Cuba, Bejucal	"	8'28	82'98	9'44	0'09	100'79	
California, Waldorf	Hard	6'47	82'77	10'62	0'35	100'21	
" La Patera	"	6'23	83'30	9'88	0'70	100'11	
Trinidad Lake	"	6'16	82'33	10'69	0'81	99'99	
Tobago	Pitch	6'16	83'37	10'54	0'50	100'57	
Turkey	Hard	6'14	83'98	9'19	0'09	90'40	
Montana, hardened	"	5'31	—	—	—	—	
California, Santa Maria	Cheesy	5'69	82'79	11'58	0'48	100'54	
Trinidad, land	Hard	4'98	83'65	10'76	0'45	99'94	
France, South	"	4'78	82'07	11'58	1'56	99'99	
Venezuela, Bermudez, hard	Medium	3'93	—	—	0'75	—	
Sicily, rock	"	3'69	84'95	11'34	0'33	100'31	
Kentucky	"	3'39	84'16	11'52	1'58	99'65	
Texas, Rio Grande	"	3'28	—	—	—	—	

Malthas Rapidly Hardening.

Venezuela, Bermudez, Maltha	—	5'87	—	—	—	—
Montana, Maltha	—	3'92	—	—	—	—

Matthas.

Venezuela, Pedernales	2.20	—	—	—	—	—
Ohio, residuum	2.26	—	—	—	—	—
Trinidad, Guayaguayare	2.00	—	—	—	—	—
Indian Territory, Chickasaw	1.98	85.65	12.37	—	—	100.00
Trinidad, Union Maltha	1.75	—	—	—	—	—
Mexico	1.48	—	—	—	—	—
Indian Territory, Ardmore	1.47	87.40	11.05	—	0.65	100.57
California, Asphalt	1.40	84.31	12.41	—	1.35	99.47
" Alcatraz	1.32	85.72	11.83	—	1.21	100.08
Texas, St. Joe	1.13	87.27	11.79	—	0.23	100.42
Pennsylvania, residuum	0.60	87.44	12.50	—	—	—

Gilsonite, Elaterite, Albertite, Ozokerite.

Wurtzelite, Utah..	5.83	80.00	12.23	—	1.78	99.84
Gilsonite, Utah	1.79	89.28	8.66	—	0.79	100.52
Albertite, New Brunswick..	1.20	85.53	13.20	—	0.42	100.35
Ozokerite, soluble in alcohol	0.00	85.25	15.09	—	0.00	100.34

Artificial Asphalts.

Pittsburg flux	4.10	83.05	10.58	—	2.38	99.84
Sludge asphalt	4.87	87.17	7.94	—	0.00	99.98
Byerlite, hard	—	89.88	10.12	—	—	100.00

This substance, being formed largely from the saturated paraffin hydrocarbons, is, in consequence, quite different from the true asphalts in many of its properties. It will not pull into strings on melting and is very short.

The sludge asphalt made from the sludge tar of the kerosene refiners is much more like natural asphalt, as it pulls out to strings, like the latter melts easily, and has the general physical properties of ordinary asphalts. In this case these properties are due to the fact that the artificial product is derived from the unsaturated hydrocarbons of the petroleums, rather than the paraffins, as with Pittsburg flux, and to the presence of sulphur in the reaction which takes place, nearly 5 per cent. being left in the substance.

In the hard Byerlite an artificial asphalt is found which is of still different origin, being produced by the action of oxygen on the heavy oils of petroleum. In this way a substance is obtained in which sulphur is absent, and has also taken no part in the formation. It seems to bear the same relation to the other artificial asphalts that gilsonite does to glance pitch of asphaltic origin, and has many similar physical properties.

The results of Mr. Richardson's examinations, therefore, seem sufficient to show that sulphur plays an important part in the hardening of asphalts, although, like the artificial asphalts, some natural bitumens occur which have become hardened in another way, and, perhaps, by oxygen. In connection with the discussion of the nature and origin of natural asphalt, these facts seem to be of considerable interest.

Action of Acids on Asphalts Quantitatively Determined.

In the use of sulphuric acid of different strengths as a reagent for the treatment of asphalt oils, the action was such

that it seemed of interest to determine quantitatively, as far as possible, what this action would be upon the entire bitumen. These results, it seemed, would prove of relative interest at least, and probably of technical value. Of course, an asphalt cannot be directly treated with acid successfully. To do this satisfactorily the bitumen is dissolved in a solvent, such as naphtha, which is not strongly affected by the acid. Naphtha was selected for the purpose, because, although the entire bitumen of most asphalts is not soluble in it, the part that is not soluble may be neglected, as it would be entirely removed by the action of acid. It seemed, therefore, only necessary to study the action of the acid on the naphtha solution. This has been done with various asphalts, and with acids of various strengths, in the following manner.

A weighed amount—2 grms.—of any asphalt was treated with boiling 88 deg. naphtha until extracted, and then diluted to 250 c.c. It was then shaken twice for exactly five minutes at ordinary temperatures, with two portions of the sulphuric acid. After this treatment the naphtha solution with its remaining bitumen was washed once with water, then with alkali, and finally with water, the naphtha solution being then evaporated in a weighed platinum dish, dried for ten minutes at 100 deg. Cent., and the bitumen unacted upon by acid determined. Following are the results, including some obtained with a similar treatment of heavy petroleum oils from Pennsylvania, Ohio, and Russia for comparison.

It is, of course, found that the heavy petroleum oils from Pennsylvania, Ohio and Russia are the least affected, as they are largely made up of paraffin hydrocarbons and naphthenes not readily acted on by acid in dilute naphtha solutions. Among the asphalts the malthas are least

PER CENT. OF NAPHTHA-SOLUBLE BITUMEN IN ASPHALT AND HEAVY OILS UNACTED UPON BY ACID.

Name.	Total bitumen.	Naphtha soluble bitumen.	Percentage of total bitumen thus soluble.*	Percentage of naphtha-soluble bitumen unacted on by					Fuming $H_2SO_4 + SO_3$.
				25 per cent. NaOH.	3 to 1 H_2SO_4 .	2 to 1 H_2SO_4 .	1 to 1 H_2SO_4 .	Conc. H_2SO_4 .	
				84.8	71.8	67.1	64.4	54.4	
California consolidated..	66.9	47.78	71.40	84.8	71.8	67.1	64.4	54.4	4.0
Bermudez lake	96.5	67.27	69.70	80.8	80.7	80.9	78.8	38.9	19.1
Trinidad lake	57.1	34.55	60.50	90.1	89.8	87.0	79.3	38.9	35.3
Alcatraz maltha (1894)..	97.9	85.19	87.00	100.0	93.7	88.4	87.0	64.2	35.7
Penn. residuum	100.0	95.68	95.71	97.4	95.5	95.6	95.5	89.5	53.3
Ohio residuum	100.0	97.05	97.10	99.5	94.9	93.4	93.6	86.4	62.6
Russian residuum	100.0	100.00	100.00	100.0	93.6	92.7	92.2	79.3	68.8

Or, calculated on another basis:—

PER CENT. OF TOTAL BITUMEN REMOVED BY RE-AGENTS.

California consolidated..	—	—	—	—	48.7	52.1	54.0	61.2	97.1
Bermudez lake	—	—	—	—	43.5	43.6	45.1	72.8	86.7
Trinidad lake	—	—	—	—	45.7	47.3	52.1	76.5	78.6
Alcatraz maltha	—	—	—	—	17.7	23.1	24.3	44.2	69.6
Penn. residuum	—	—	—	—	8.5	8.5	8.6	14.4	49.0
Ohio residuum	—	—	—	—	7.9	9.4	9.2	16.2	39.2
Russian residuum	—	—	—	—	6.4	7.3	7.8	20.7	31.2

* More thorough extraction would have increased these percentages in some cases.

attacked by common acid, showing that they lie between the heavy petroleum oils and the asphalts, while the harder the asphalt, the smaller is the per cent. of bitumen unacted upon by acids, corresponding relatively to the amount soluble in naphtha.

Effect of Heat on Mineral Oils.

In a recent article (*Municipal Engineering*, August, 1897) Mr. Richardson has shown that there is a difference in the way different asphalts, malthas, and heavy petroleum oils are affected by being maintained at a high temperature for a number of hours. While all of them volatilise something when heated to the conventional temperatures of the asphalt industry—325 deg. and 400 deg. Fah. for seven hours—there is a very considerable difference in the appearance of the residues after heating.

Petroleum residuum of the kind best suited for the asphalt-paving industry, and made especially for it, may be heated for seven hours at 400 deg. Fah. and lose but 2 to 5 per cent. of volatile oil, while the residue will still continue to flow at ordinary temperatures. Other petroleum residuums may lose a much higher percentage of oil up to 15 per cent., and leave a residue, which will not flow owing to the amount of scale paraffin it contains; but in neither case is the resulting residue solid and brittle. Many natural heavy tarry oils, known as malthas or soft asphalts, will, however, not only lose a large amount of volatile oil when heated to 325 deg. or 400 deg. Fah., up to 20 or 30 per cent., but they will leave behind a residue which is a brittle glance pitch, showing the very different nature of the hydrocarbons of which it is composed, and those making up the residuum of ordinary Eastern petroleum. Malthas of a medium character are also found, so that it is a very useful

means of determining the character of any heavy mineral oil to examine it in this way.

It appears that there is a very decided difference in the character of the bitumens found in various asphalts, malthas, and heavy petroleum oils, which is shown in several ways by the preceding methods of treatment. The amount of total bitumen which is soluble in 88 deg. naphtha is, in the first place, indicative of how far the action of condensation and polymerisation has gone on, and of how large a part of the bitumen has been converted into the hard and brittle form. For example, in the heavy petroleum oils and malthas the larger portion of the bitumen is soluble in naphtha, while in the asphalts this may be reduced to 60 per cent. or less, and in the glance pitches to 40. It is possible, therefore, to gain considerable assistance in judging of the nature of a new asphalt by such a determination, or at least, as to some of its properties of technical interest. In another way the determination of the amount of the hydrocarbons soluble in naphtha, which are unacted upon by acid, helps to show the nature of the substances making up this part of the bitumen. And again, the stability of the hydrocarbons is shown by their power of resisting high temperatures with little loss and without change in their physical properties, and especially their consistency.

Perhaps none of the tests which have been given possess any actual scientific merit, but they at least enable us, when they are combined, to compare different asphalts, and to judge of their technical value. It must also be remembered that the results have no absolute value, and can be used only as a relative means of comparison, since the solvents and conditions may vary from time to time. In making examination of this kind, therefore, it is desirable that a well-known asphalt, maltha, or oil should be subjected to a

parallel analysis, alongside of the material under investigation.

Conclusions as to the Nature and Origin of Asphalt.

The natural bitumen, which is known as asphalt, is composed, as far as we have been able to learn, of saturated and unsaturated di-cyclic, or polycyclic, alicyclic hydrocarbons and their sulphur derivatives, with a small amount of nitrogenous constituents. Asphalt may therefore be defined as any hard bitumen composed of such hydrocarbons and their derivatives, which melts on the application of heat to a viscous liquid ; while a maltha or soft asphalt may be defined as a soft bitumen, consisting of alicyclic hydrocarbons, which, on heating, or by other natural causes, becomes converted into asphalt. The line between the two classes cannot be sharply drawn.

Asphalts are distinguished by the large amount of sulphur they contain, and it is to its presence that many of the important characteristics and perhaps, in part, the origin of this form of bitumen is due. The soft asphalts or malthas contain much less sulphur than the harder ones, or if the former are rich in sulphur, they are then in a transition stage and will eventually become hard. But a small portion of the constituents of a hard asphalt are volatile even in vacuo, but they can be separated by solvents into an oily portion, which is soft, or softens readily when heated, and a harder portion, which does not melt by itself without decomposition, and is a brittle solid, but soluble in the oily or softer portion. The harder and least soluble portion always contains the larger part of the sulphur. It seems, therefore, that sulphur is the effectual hardening agent of natural asphalts, in the same way that it is of artificial asphalts which

are produced by heating a soft natural bitumen with sulphur.

The portion of asphalt soluble only in chloroform or carbon bisulphide is entirely absorbed by sulphuric acid, showing that it contains no paraffins or saturated hydrocarbons.

That portion which is soluble in naphtha can be, to a great extent, volatilised in vacuo. The lighter constituents are found to be hydrocarbons of at least two classes—those absorbed or converted into tars by sulphuric acid, and those but slightly acted upon by it, together with sulphur compounds partly precipitated by mercuric chloride and partly recoverable from their combination with sulphuric acid by steam.

The hydrocarbons absorbed by sulphuric acid and converted into tars by it may belong to several series, less saturated than the C_nH_{2n} , and it is probable that some of them or their derivatives are unsaturated polycyclic alicyclic compounds. Nothing further is known of them at present. Those not easily acted upon by acid are of very high density and refractive index for their boiling point and molecular weight, and correspond in no way with any saturated or open-chain hydrocarbons. They are evidently, as has been shown, saturated polycyclic hydrocarbons similar to the naphthenes, but having an even greater density than the latter, as far as they are known, for the same molecular weight. The amount of these comparatively stable hydrocarbons unacted upon readily by sulphuric acid varies in different asphalts from comparatively little to 30 per cent. or more. In the malthas there is a still larger proportion. The greater part of an asphalt bitumen consists, however, of unsaturated and unstable bodies liable to constant change, while even the most stable constituents,

the saturated alicyclic hydrocarbons, are slowly polymerised on standing.

Origin of Asphalt.

Asphalt is in the process of formation to-day. It plainly does not originate as such, but is a secondary product, resulting from the transformation of suitable lighter forms of bitumen, malthas, or even thinner oils, into harder bitumen by condensation and polymerisation, a reaction in which sulphur and probably sulphates seem to take an important part.

Asphalt is probably derived from oils consisting of alicyclic hydrocarbons, although the condensation of paraffin hydrocarbons by sulphur or of a mixture of saturated and unsaturated hydrocarbons in the same way would yield similar products, as we have seen in the artificial Pittsburg flux. Polymerisation would, perhaps, take place more readily in the unsaturated series.

No high temperatures seem necessary in nature for the change once initiated, as it is going on to-day in the Trinidad pitch lake and in Venezuela. Gas is commonly evolved and is largely hydrogen sulphide, the natural result of the condensation of hydrocarbons by sulphur. Carbonic acid accompanies it, which would seem to point to a reaction between hydrocarbons and inorganic sulphates and the latter as the source of the sulphur in the bitumen of asphalts. This theory is based on the presence of sulphates in the water emulsified with Trinidad pitch, and in the California shales, while there is quite as much hydrogen sulphide given off by the Bermudez asphalt which contains no water or sulphates as it originates.

Whether its origin is in sulphates or inorganic sulphides, or whether, as suggests itself, it is derived from the same

substances, either animal, vegetable, or inorganic, from which the hydrocarbons themselves are formed, it is plainly an important factor in the production of asphalt, and especially the harder ones.

It seems justifiable, therefore, to suggest that where certain mineral oils, composed of alicyclic hydrocarbons, originate under such circumstances as to be subjected to conditions favourable to condensation and polymerisation, or to the action of sulphur or sulphates, asphalt will be formed, not necessarily immediately, but in the course of time.

For the primary origin of asphalt one must, of course, go back to that of petroleum. This subject has lately been discussed at length, and both experiments and arguments brought forward in favour of the animal or vegetable origin of petroleum or its inorganic derivation.

The presence of nitrogen in all the asphalts examined may be considered, by those advocating the animal or vegetable origin of petroleum, as confirming their views, but it seems possible also to account for it from an inorganic source.

The theory of the general origin of all mineral bitumen in a base of unsaturated hydrocarbons suggested by Heusler, seems to be supported by the unsaturated nature of most of the constituents of the asphalts and malthas; but on the other hand, the formation of saturated hydrocarbons from the unsaturated oils of Trinidad asphalt by the action of aluminium chloride has not yet been accomplished.

The thermal origin of the bitumen of Trinidad lake asphalt is strongly suggested by the presence of a thermal water, containing borates and iodides, in an emulsion of definite composition with bitumen and mineral matter,

while the transportation of bitumen from place to place by means of springs is illustrated by the mineral spring which rises at the centre of the Trinidad lake with the freshly issuing pitch, and to the collection of bitumen in pockets, even under the sea where springs break out. In no case, however, have any signs of more than normal temperatures been observed in any of these springs. Steam, therefore, if an element in the movement of this bitumen, must be associated with it at such a point as to indicate a thermal origin of the bitumen itself.

Finally, the theory of Ochsenius, that the formation of petroleum is to be attributed to the action of strong salt solutions on organic matter, might also be considered as strengthened by the presence of such a strong brine as accompanies the emulsion of soft asphalt in the Trinidad lake.

Chinese and Egyptian Bitumen.—Boussingault* examined the bitumen from the so-called burning springs of Ho-Tsing, in the Province of Szu-Tchuan, China, where, in a region having an area of fifty square miles, several thousand springs exist, whence proceed inflammable gases at a high velocity, accompanied by bitumen and brine. At ordinary temperatures the bitumen is in a fluid condition, but when cooled naphthalene separates out. The solid and liquid constituents of the bitumen separated by cooling and filtration have the following composition:—

	Carbon.	Hydrogen.	Oxygen.	Nitrogen.
	Per cent.	Per cent.	Per cent.	Per cent.
Solid	82·85	13·09	4·06	0·00
Liquid	86·82	13·16	0·00	0·02

The same author gives the following results of the exami-

* *Journal of the Society of Chemical Industry*, Vol. i.¹, page 159.

nation of samples of asphaltum respectively from Egypt and from the Dead Sea :—

	Carbon.	Hydrogen.	Oxygen.	Nitrogen.
	Per cent.	Per cent.	Per cent.	Per cent.
Egypt.. ..	85.29	8.24	6.22	0.25
Dead Sea	77.84	8.93	11.53	1.70

Since the appearance of the preceding paper further investigations have resulted in showing, according to Mr. Richardson, that the rate of influx of asphalt into the pitch lake is strongly influenced by the rapidity with which material is removed from it. In two years, when the pitch was taken out in larger quantities than usual, the influx was greater than in ordinary years; that is to say, the rapid removal of pitch produces a more sudden removal of the pressure on the source of supply, and a greater evolution of fresh pitch takes place. Levels of the lake's surface are still being taken yearly, and most interesting information is continually being obtained.

Investigations as to whether the finest inorganic matter, not heretofore considered removable from the bitumen of Trinidad asphalt, is actually in combination with the latter, as has been claimed, have shown that all inorganic matter can be filtered out by passing a solution of the bitumen in carbon bi-sulphide through a Pasteur filter, and that on evaporation of the solvent the bitumen is quite free from even a trace. The mineral matter is therefore in no way in combination with the hydrocarbons of the asphalt or their derivatives.

Investigations of the lighter hydrocarbons in the bitumen of Trinidad asphalt and of their sulphur and nitrogen derivatives have been continued, and the characteristics of these substances have been compared with those of the

similar constituents of the heavy petroleum of California, and the asphalt deposits of that State being now well understood, it becomes evident that the origin of the bitumen in Trinidad lake asphalt is in petroleum.



THE END

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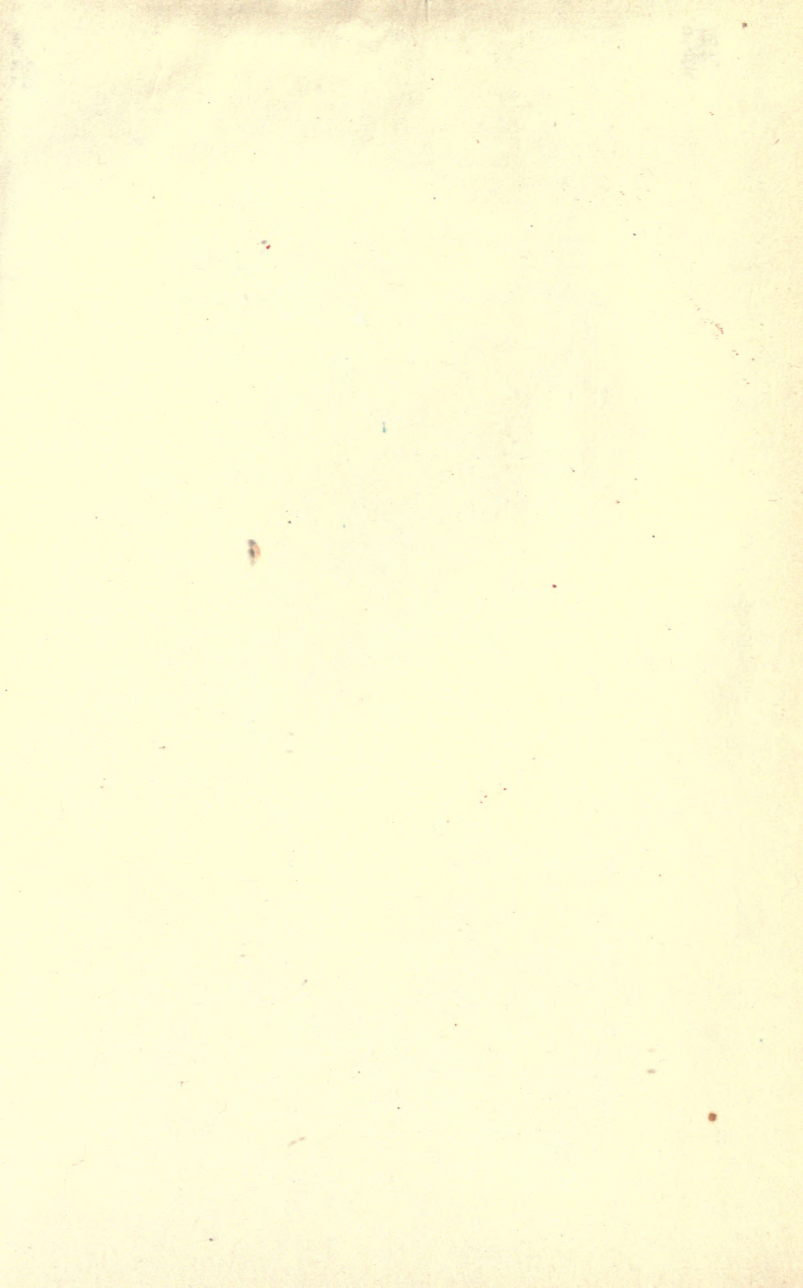
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